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Honeywell's Docket No. 30-4874 - 4690
Practitioner's Docket No. H9910-0305   *PATENT*

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: James Kweeder, et al.

Application No.: 09/468668   Group No.: 1761
Filed: December 21, 1999   Examiner: Robert A. Madsen
For: Prilling Method

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P.O. Box 1450
Alexandria, VA 22313-1450

TRANSMITTAL OF RESPONSE TO NOTICE OF NON-COMPLIANT APPEAL BRIEF
(PATENT APPLICATION--37 C.F.R. § 41.37)

1.   Transmitted herewith is the RESPONSE TO THE NOTICE OF THE NON-COMPLIANT APPEAL BRIEF in this application, with respect to the Notice of Appeal filed on December 2, 2005.

2.   STATUS OF APPLICANT

This application is on behalf of other than a small entity.

CERTIFICATION UNDER 37 C.F.R. §§ 1.8(a) and 1.10*
*(When using Express Mail, the Express Mail label number is **mandatory**;*
Express Mail certification is optional.)

I hereby certify that, on the date shown below, this correspondence is being:

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37 C.F.R. § 1.8(a)	37 C.F.R. § 1.10*
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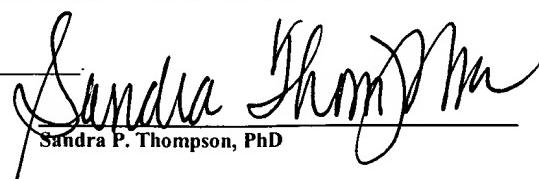
Sandra P. Thompson, PhD

Date: _5/30/2006_

Transmittal of Appeal Brief--page 1 of 2

3. **FEE FOR FILING APPEAL BRIEF**

Pursuant to 37 C.F.R. §41.20, the fee for filing the Appeal Brief is:

other than a small entity $500.00

Appeal Brief fee due $500.00

4. **EXTENSION OF TERM**

The proceedings herein are for a patent application and the provisions of 37 C.F.R. § 1.136 apply.

Applicant believes that no extension of term is required. However, this conditional petition is being made to provide for the possibility that applicant has inadvertently overlooked the need for a petition and fee for extension of time.

5. **TOTAL FEE DUE**

The total fee due is:

Appeal brief fee $500.00 (Already charged to Account)
Extension fee (if any) $0.00

**TOTAL FEE DUE $0.00**

6. **FEE PAYMENT**

There should be no fee payment due at this time.

7. **FEE DEFICIENCY**

If any additional extension and/or fee is required, and if any additional fee for claims is required, charge Deposit Account No. 500977.

Date: __5/30/2006__
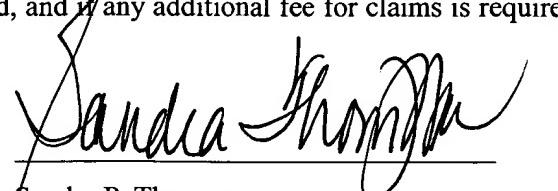
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Application No.: 09/468,668   Group No.: 1761

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For: **PRILLING METHOD**

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RESPONSE TO NOTICE OF NON-COMPLIANT APPELLANT'S BRIEF UNDER 37 CFR § 41.37

This Response to the Notice of Non-Compliant Appeal Brief is filed on Tuesday, May 30, 2006. It was originally due on Sunday, May 28th, and therefore is timely filed on Tuesday, May 30.

This revised brief contains the following items under the headings in the order here indicated:

APPELLANTS BRIEF UNDER 37 CFR § 41.37

REAL PARTY IN INTEREST

RELATED APPEALS AND INTERFERENCES

STATUS OF THE CLAIMS

STATUS OF AMENDMENTS

SUMMARY OF CLAIMED SUBJECT MATTER

GROUNDS OF REJECTION TO BE REVIEWED ON APPEAL

ARGUMENT

CLAIMS APPENDIX

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RELATED PROCEEDINGS APPENDIX

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REAL PARTY IN INTEREST

The real party in interest is the assignee, Honeywell International Inc. (see Reel/Frame No. 010558/0960, Recorded on January 14, 2000)

RELATED APPEALS AND INTERFERENCES

There are no other appeals or interferences in this matter known to appellant.

STATUS OF THE CLAIMS

There are 17 claims in this case.

Claims 11-13 were withdrawn in the Request for Continued Examination dated November 5 2002 as being drawn to a non-elected invention.

Claim 14 was canceled in the Request for Continued Examination dated November 5 2002.

Claims 1-10 and 15-17 are pending.

STATUS OF AMENDMENTS

There have been no amendments filed subsequent to the advisory action received on November 1, 2005 in this matter. This advisory action was in response to a Response After Final filed on August 11, 2005.

SUMMARY OF THE CLAIMED SUBJECT MATTER

This invention describes prilling methods, specifically methods of prilling shear-thinnable mixtures of a meltable first component and a second component using mechanical agitation in a prill head to shill thin the mixture. (See page 6, lines 18-end and page 7, lines 1-5). The key to the invention is the introduction of agitation into the prill head, which introduces shear-thinning to the high viscosity molten mixture. (See page 6, lines 18-25). Agitation in the prill head can be introduced in a number of ways, but a preferred agitation device is one where essentially the entire liquid volume in the prill head is swept by the agitator. (See page 6, lines 26-end and page 7, lines 1-5).

The methods include (see page 6, lines 5-25):

a)   providing a molten first component; (see page 8, lines 6-20)

b)   mixing at least a second component with said molten first component; (see page 8, lines 6-30)

c)   reacting said components at a temperature and for a time sufficient to form a shear-thinnable mixture having a viscosity, whereby the viscosity decreases with increased shear rate (see page 8, lines 30-37; page 9, lines 10-26; page 5, lines 26-end and page 6, lines 1-5);

d)   mechanically agitating said shear-thinnable mixture by an agitator in a prill head, wherein essentially the entire liquid volume in said prill head is swept by said agitator to shear thin said shear-thinnable mixture; and permitting said shear-thinned mixture to flow through holes in said prill head under the influence of a force selected from the group consisting of static pressure and centrifugal force. (see page 6, lines 26-38, page 7, lines 1-11, Inventive Examples 1 and 2 on pages 11 and 12)

GROUNDS OF REJECTION TO BE REVIEWED ON APPEAL

Claims 1-6 are rejected under 35 USC §103(a) as being unpatentable over Hoogendonk (US 3083406) in view of Holland et al (1995), Hanke et al (US 5466281) and Otsuka et al (US 3529326).

Claims 7-10 are rejected under 35 USC §103(a) as being unpatentable over Hoogendonk (US 3083406) in view of Holland et al (1995), Hanke et al (US 5466281) and Otsuka et al (US 3529326), further in view of Frenken et al (US 3,988,398).

Claim 15 is rejected under 35 USC §103(a) as being unpatentable over Hoogendonk (US 3083406) in view of Holland et al (1995), Hanke et al (US 5466281) and Otsuka et al (US 3529326) as applied to claims 1-6 above, further in view of Bassetti et al (US 5378259).

Claims 16-17 are rejected under 35 USC §103(a) as being unpatentable over Hoogendonk (US 3083406) in view of Holland et al (1995), Hanke et al (US 5466281) and Otsuka et al (US 3529326) as applied to claims 1-6, further in view of Stengel (US 3021207). The Applicant respectfully disagrees, especially in view of the amendments presented herein.

ARGUMENT

ISSUE NO. 1 - §103 (A) REJECTION OF CLAIMS 1-6

Claim 1 recites:

"A method to prill a shear-thinnable mixture comprising the steps of:

a)   providing a molten first component;

b)   mixing at least a second component with said molten first component;

c)   reacting said components at a temperature and for a time sufficient to form a shear-thinnable mixture having a viscosity, whereby the viscosity decreases with increased shear rate;

d)   mechanically agitating said shear-thinnable mixture by an agitator in a prill head, wherein essentially the entire liquid volume in said prill head is swept by said agitator to shear thin said shear-thinnable mixture; and permitting said shear-thinned mixture to flow through holes in said prill head under the influence of a force selected from the group consisting of static pressure and centrifugal force.

THIXOTROPIC VERSUS SHEAR-THINNABLE

The Examiner has contended throughout the prosecution of this application that the terms "thixotropic" and "shear-thinnable" mean the same thing and are interchangeable. The terms *thixotropic* and *shear-thinnable*, while related, are not interchangeable and not understood in the field to mean the same thing.

Reference is made to Holland (1995) and Hanke (US 5,466,281) to support the assertion that thixotropic and shear-thinning are synonymous terms. The applicant strenuously continues to disagree with that assertion. Holland clearly defines the terms to be different (as expounded in

the James Kweeder (inventor) declaration filed on August 8, 2005). Shear-thinning fluids (sometimes referred to as *psedoplastic*) are those fluids whose viscosity decreases with increasing shear-rate. Thixotropic fluids, conversely, are those whose viscosity decreases with time under constant shear rate. Oldshue's classic text (attached herein for immediate reference) on fluid mixing supports this distinction.

Unfortunately, confusion has arisen on some of this terminology. The proper term for shear-thinning, *pseudoplastic*, has also come to mean a particular behavior of polymer melts (a Newtonian, non-Newtonian, and then again Newtonian viscosity). This ambiguity is nicely discussed in Holland. Consequently, the term shear-thinning has come to be used for the generic behavior of decreasing shear with increased shear rate. Examination of the Holland reference, particularly figures 1.20 and 1.21 (and the referencing text) illustrate the difference. *Shear-thinnable* refers to those liquids in which the viscosity decreases with increasing shear rate independent of time. *Thixotropic*, alternatively, describes a fluid in which the viscosity decreases with time while at constant shear-rate. While it is possible that a particular fluid be both thixotropic and shear-thinning (as illustrated in figure 1.21 of Holland), it is not required. A thixotropic viscosity can be independent of shear-rate (that is, Newtonian with respect to shear-rate but non-Newtonian with respect to time).

Further, it is certainly apparent that shear is required to bring-forth the thixotropic behavior of those fluids. Consequently, those inadequately skilled in the art have from time to time used the terms interchangably. *That usage – however - is grievously erroneous* and has been discussed by the Applicants in several documents submitted already in the case.

This lamentable ambiguity in terminology necessitates careful workers to define the meaning of their usage, as has been done in the current application. In this application, shear-thinning has been defined to mean decreasing viscosity with increasing shear-rate (see page 5, lines 33-35). Experimental evidence is also presented in the application to illustrate the meaning absolutely.

However, no such ambiguity exists with the term thixotropic. Thixotropic is defined strictly as time-dependent viscosity. While Hanke may chose to treat the terms "shear-thinnable" and "thixotropic" interchangeably, the Applicant continues to point out that engineering literature (typified by Holland and Oldshue) most definitely does not support that usage.

Holland (and others) do point out that often thixotropic fluids are also shear-thinning (pseudoplastic) fluids. This common situation of time and shear-dependent viscosity gives rise to statements (like in Holland) of "in practice, thixotropic fluids are also shear-thinning". However, such statements do not guarantee that a thixotropic fluid is indeed shear-thinning. Further, such generalizations do not in any way change the definitions of the terms thusly erroneously rendering time-dependent and shear-dependent to mean the same thing.

A consequence of equating time and shear-dependence is the contention that shear is shear. Nothing can be further from the truth. As previously discussed in the James Kweeder declaration, shear-thinning requires device design with an eye toward generating the necessary shear-rate to achieve the desired reduction in viscosity. Thixotropy, conversely, requires attention to the necessary time to achieve viscosity reduction. Of course, a device designed for a fluid that is both shear-thinning and thixotropic will need to address both needs.

An excellent illustration of such sweeping over-extrapolation is the Examiner's analysis of Hoogendonk (US 3,083,406). In the examination, the assumption is made by the Examiner that since shear is being applied to the fluid at the roller-wall interface, consequently, the whole of the liquid volume is subjected to that shear and thusly capable of flowing under the centrifugal forces of the rotating prill cup. Implicit to that assumption is that the benefits of shear (argued as thixotropic by Hoogendonk) is realized throughout the system so long as it's available somewhere, anywhere in the system.

Further, despite the casual statement by Hoogendonk, it is by no means obvious that its inventive device functions because of thixotropy. Indeed, the '406 inventive device is an improvement on a previous device (US 3,055,049 on which Hoogendonk is also an inventor), which relies on a non-roller implementation to keep the inner walls of the prilling device clean.

US 3,055,049 makes no claims nor arguments for the device advantageously modifying the fluid viscosity. Changing-out the fixed scraper for a roller-scraper does not modify the shear-mixing properties of the inventive device in any way. Consequently, the inventive device in US 3,083,406 is likely to be functional for the same reason that the inventive device was functional in US 3,055,049.

Further, as one skilled in the art and in light of definitively pressure-pumping devices disclosed in Frenken (US 3,988,398) and DE 2,355,660 (attached for immediate reference), one can argue that the inventive device in US 3,083,406 is functioning as a positive displacement, peristaltic pump that merely forces the reluctant fluid through the prilling orifice. Hoogendonk furthers enhances the positive displacement pump argument by then proposing pins on the rolling device to supplement peristaltic pumping action with a piston pumping action directly through the prilling orifice.

In US 3,083,406, Hoogendonk makes exactly one reference to thixotropy providing neither reference, experimental data, nor examples to support that argument. Given the thin zone of shear (the clearance between the roller and the wall) and other arguments for operability (US 3,055,049, US 3,988,398, DE 2,355,660), it is not obvious at all that thixotropy is responsible let alone that the inventive device is effective with shear-rate viscosity dependent fluids.

The difference between *shear-thinning* and *thixotropic* necessitates different approaches to device design. While exploiting either behavior requires shearing the material, it does not follow that one device suitable for one behavior will necessarily function as anticipated with the other behavior. In particular, *shear-thinning* usually requires that a particular shear-rate be achieved to accomplish the targeted viscosity. Consequently, it is necessary to specify that adequate shear-rate by the combination of shear device geometry and device velocity. *Thixotropy*, conversely, requires that shear be maintained for a specified time period. A device designed for shear-thinning may not sustain shear sufficiently long to process a thixotropic material and a device designed for thixotropy may not achieve adequate shear to process a shear-thinning material.

Hoogendonk Reference

Hoogendonk states in Column 1, lines 54-65 that: "In accordance with the present invention it has been found that prills of good quality can be obtained if the melt is sprayed from a reservoir which has a scraper arrangement including a rotary element rolling along the upright wall of the reservoir while the latter rotates. Owing to the rolling movement of the said element the inner wall of the reservoir remains clean and the spray openings do not become clogged. Due to the thixotropic properties of the melts to be sprayed, the shearing stresses produced by the rolling movement of the rotary element causes the melts to remain sufficiently fluid so that no solid material will deposit on the wall or in the spray openings."

Hoogendonk also states in Column 1, lines 35-42 that: "It is therefore an object of the present invention to provide an improved prilling device which prevents clogging of the spray openings and which has a relatively long life. A further object of the present invention is to provide an improved prilling device which utilizes a rolling member for preventing clogging of the spray openings." The assertion that the melts are fluid as based on thixotropic properties should be questioned. It has been asserted in the Kweeder Declaration filed on August 2, 2005 in this case that it may be the pressure created by the rollers, similar to the action of a positive peristaltic pump (see Exhibit B of this Declaration), causing the melts to prill easily over conventional methods used. In addition, if in fact the melts are fluid based on thixotropic properties, then the only part of the melt that is thixotropic is that part of the melt that is between the inner wall and the rollers. There is absolutely no teaching or suggestion in Hoogendonk that any portion of the remaining melt, such as that portion of the melt in the middle of the apparatus, is being swept by the rollers or thixotropic.

Hoogendonk mentions thixotropic fluids briefly without any discussion on these types of fluids or the shear-rate and/or time requirements when working with thixotropic fluids. Hoogendonk also does not suggest to one of ordinary skill in the art that there might be some benefit to sweeping and/or agitating the entire liquid volume in the prill head to shear thin a

shear-thinnable mixture. If the agitator action of Hoogendonk does in fact work, it would be limited to the shear zone between the roller and the wall. The agitator action would not affect the entire liquid volume in the prill head.

Hoogendonk discloses a prilling device with an integral "agitator" that is specifically configured to roll along the inside wall of the prilling device for the specific purpose of keeping the prilling orifices "clear". One embodiment includes pins on the rolling device to mechanically clear the holes, the other is silent in whether a smooth roller or something textured is used to enhance clearing. (see Column 1, lines 59-66) But there is no teaching or suggestion that there is any benefit to using rollers to agitate or sweep the entire liquid volume of the reservoir, as mentioned in claim 1 of the above-referenced patent application.

Otsuka Reference

The Examiner should remove Otsuka et al. as a 35 USC §103(a) reference in this case. The Otsuka reference is filled with references to how difficult it is to take the molten materials described in Otsuka and process them by conventional prilling methods, including those that utilize agitators. (see Columns 5-6) Therefore, in order to combat the problems seen when incorporating the molten materials of Otsuka, Otsuka engineers using a series of mesh screens to break up and disperse the molten materials. After a fair reading of Otsuka, one of ordinary skill of the art would understand how to make molten materials containing nitrogen and potassium or phosphorus - but there is absolutely no suggestion or teaching that these molten compounds can be utilized by any conventional or modified prilling methods other than those described in the Otsuka reference, especially after reading Columns 5 and 6 of the reference. Therefore, combining this reference with another reference or references that describe prilling methods unlike those in Otsuka would be considered improper combination based on hindsight. The Applicant has respectfully requested that the Examiner review the Otsuka reference paying close attention to Columns 5 and 6 and withdraw this reference as a relevant reference in this matter.

Holland and Hanke References

The Holland and Hanke references merely disclose thixotropic and shear-thinning materials. There doesn't appear to be anything in either the Holland reference or the Hanke reference that cures the deficiencies of the Otsaka reference, the Hoogendonk reference or the Frenken reference, in combination with one or all of them, that would lead someone in the field of fertilizer chemistry and fertilizer production to prill a shear-thinnable mixture.

ISSUE NO. 2 - §103 (A) REJECTION OF CLAIMS 7-10 AS APPLIED TO CLAIMS 1-6

Claim 1 recites:

"A method to prill a shear-thinnable mixture comprising the steps of:

a)   providing a molten first component;

b)   mixing at least a second component with said molten first component;

c)   reacting said components at a temperature and for a time sufficient to form a shear-thinnable mixture having a viscosity, whereby the viscosity decreases with increased shear rate;

d)   mechanically agitating said shear-thinnable mixture by an agitator in a prill head, wherein essentially the entire liquid volume in said prill head is swept by said agitator to shear thin said shear-thinnable mixture; and permitting said shear-thinned mixture to flow through holes in said prill head under the influence of a force selected from the group consisting of static pressure and centrifugal force.

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Reference is made to Holland (1995) and Hanke (US 5,466,281) to support the assertion that thixotropic and shear-thinning are synonymous terms. The applicant strenuously continues to disagree with that assertion. Holland clearly defines the terms to be different (as expounded in the James Kweeder (inventor) declaration filed on August 8, 2005). Shear-thinning fluids

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Unfortunately, confusion has arisen on some of this terminology. The proper term for shear-thinning, *pseudoplastic*, has also come to mean a particular behavior of polymer melts (a Newtonian, non-Newtonian, and then again Newtonian viscosity). This ambiguity is nicely discussed in Holland. Consequently, the term shear-thinning has come to be used for the generic behavior of decreasing shear with increased shear rate. Examination of the Holland reference, particularly figures 1.20 and 1.21 (and the referencing text) illustrate the difference. *Shear-thinnable* refers to those liquids in which the viscosity decreases with increasing shear rate independent of time. *Thixotropic*, alternatively, describes a fluid in which the viscosity decreases with time while at constant shear-rate. While it is possible that a particular fluid be both thixotropic and shear-thinning (as illustrated in figure 1.21 of Holland), it is not required. A thixotropic viscosity can be independent of shear-rate (that is, Newtonian with respect to shear-rate but non-Newtonian with respect to time).

Further, it is certainly apparent that shear is required to bring-forth the thixotropic behavior of those fluids. Consequently, those inadequately skilled in the art have from time to time used the terms interchangably. *That usage – however - is grievously erroneous* and has been discussed by the Applicants in several documents submitted already in the case.

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Further, despite the casual statement by Hoogendonk, it is by no means obvious that its inventive device functions because of thixotropy. Indeed, the '406 inventive device is an improvement on a previous device (US 3,055,049 on which Hoogendonk is also an inventor), which relies on a non-roller implementation to keep the inner walls of the prilling device clean. US 3,055,049 makes no claims nor arguments for the device advantageously modifying the fluid viscosity. Changing-out the fixed scraper for a roller-scraper does not modify the shear-mixing

properties of the inventive device in any way. Consequently, the inventive device in US 3,083,406 is likely to be functional for the same reason that the inventive device was functional in US 3,055,049.

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The difference between *shear-thinning* and *thixotropic* necessitates different approaches to device design. While exploiting either behavior requires shearing the material, it does not follow that one device suitable for one behavior will necessarily function as anticipated with the other behavior. In particular, *shear-thinning* usually requires that a particular shear-rate be achieved to accomplish the targeted viscosity. Consequently, it is necessary to specify that adequate shear-rate by the combination of shear device geometry and device velocity. *Thixotropy*, conversely, requires that shear be maintained for a specified time period. A device designed for shear-thinning may not sustain shear sufficiently long to process a thixotropic material and a device designed for thixotropy may not achieve adequate shear to process a shear-thinning material.

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Hoogendonk also states in Column 1, lines 35-42 that: "It is therefore an object of the present invention to provide an improved prilling device which prevents clogging of the spray openings and which has a relatively long life. A further object of the present invention is to provide an improved prilling device which utilizes a rolling member for preventing clogging of the spray openings." The assertion that the melts are fluid as based on thixotropic properties should be questioned. It has been asserted in the Kweeder Declaration filed on August 2, 2005 in this case that it may be the pressure created by the rollers, similar to the action of a positive peristaltic pump (see Exhibit B of this Declaration), causing the melts to prill easily over conventional methods used. In addition, if in fact the melts are fluid based on thixotropic properties, then the only part of the melt that is thixotropic is that part of the melt that is between the inner wall and the rollers. There is absolutely no teaching or suggestion in Hoogendonk that any portion of the remaining melt, such as that portion of the melt in the middle of the apparatus, is being swept by the rollers or thixotropic.

Hoogendonk mentions thixotropic fluids briefly without any discussion on these types of fluids or the shear-rate and/or time requirements when working with thixotropic fluids. Hoogendonk also does not suggest to one of ordinary skill in the art that there might be some benefit to sweeping and/or agitating the entire liquid volume in the prill head to shear-thin a

shear-thinnable mixture. If the agitator action of Hoogendonk does in fact work, it would be limited to the shear zone between the roller and the wall. The agitator action would not affect the entire liquid volume in the prill head.

Hoogendonk discloses a prilling device with an integral "agitator" that is specifically configured to roll along the inside wall of the prilling device for the specific purpose of keeping the prilling orifices "clear". One embodiment includes pins on the rolling device to mechanically clear the holes, the other is silent in whether a smooth roller or something textured is used to enhance clearing. (see Column 1, lines 59-66) But there is no teaching or suggestion that there is any benefit to using rollers to agitate or sweep the entire liquid volume of the reservoir, as mentioned in claim 1 of the above-referenced patent application.

Frenken Reference

The Frenken device includes a pump impeller with the specific purpose of raising the interior fluid pressure and forcing the melt to flow through the holes. The above-referenced patent application very specifically states that they prill through a combination of static and/or centrifugal pressure and not via pressurization.

The Frenken reference, like the Hoogendonk reference, is not placing any importance or critical embodiment on sweeping and/or agitating essentially the entire liquid volume in the reservoir to shear-thin a shear-thinnable liquid. In Column 2 of the reference, lines 14-23, it is explicitly stated that the distance from the inner wall of the reservoir to the ends of the blades **is not critical**.

As a comparison, DE 2355660 teaches a cylindrical chamber with stirring blades, similar to that described in Frenken. However, the DE 2355660 points out that modifying the configuration of the chamber to be similar to the one described in the above-mentioned patent application would result in thickening, clogging of the prill holes, nonuniform product, large fraction of reject course grains and occasional large agglomerates that did not solidify in the prill

tower. Therefore, DE 2355660 teaches away from modifying the device in Frenken to arrive at the apparatus disclosed in the present application.

Otsuka Reference

The Examiner should remove Otsuka et al. as a 35 USC §103(a) reference in this case. The Otsuka reference is filled with references to how difficult it is to take the molten materials described in Otsuka and process them by conventional prilling methods, including those that utilize agitators. (see Columns 5-6) Therefore, in order to combat the problems seen when incorporating the molten materials of Otsuka, Otsuka engineers using a series of mesh screens to break up and disperse the molten materials. After a fair reading of Otsuka, one of ordinary skill of the art would understand how to make molten materials containing nitrogen and potassium or phosphorus - but there is absolutely no suggestion or teaching that these molten compounds can be utilized by any conventional or modified prilling methods other than those described in the Otsuka reference, especially after reading Columns 5 and 6 of the reference. Therefore, combining this reference with another reference or references that describe prilling methods unlike those in Otsuka would be considered improper combination based on hindsight. The Applicant has respectfully requested that the Examiner review the Otsuka reference paying close attention to Columns 5 and 6 and withdraw this reference as a relevant reference in this matter.

Holland and Hanke References

The Holland and Hanke references merely disclose thixotropic and shear-thinning materials. There doesn't appear to be anything in either the Holland reference or the Hanke reference that cures the deficiencies of the Otsaka reference, the Hoogendonk reference or the Frenken reference, in combination with one or all of them, that would lead someone in the field of fertilizer chemistry and fertilizer production to prill a shear-thinnable mixture.

ISSUE NO. 3 - §103 (A) REJECTION OF CLAIM 15

Claim 1 recites:

"A method to prill a shear-thinnable mixture comprising the steps of:

a) providing a molten first component;

b) mixing at least a second component with said molten first component;

c) reacting said components at a temperature and for a time sufficient to form a shear-thinnable mixture having a viscosity, whereby the viscosity decreases with increased shear rate;

d) mechanically agitating said shear-thinnable mixture by an agitator in a prill head, wherein essentially the entire liquid volume in said prill head is swept by said agitator to shear thin said shear-thinnable mixture; and permitting said shear-thinned mixture to flow through holes in said prill head under the influence of a force selected from the group consisting of static pressure and centrifugal force.

THIXOTROPIC VERSUS SHEAR-THINNABLE

The Examiner has contended throughout the prosecution of this application that the terms "thixotropic" and "shear-thinnable" mean the same thing and are interchangeable. The terms *thixotropic* and *shear-thinnable*, while related, are not interchangeable and not understood in the field to mean the same thing.

Reference is made to Holland (1995) and Hanke (US 5,466,281) to support the assertion that thixotropic and shear-thinning are synonymous terms. The applicant strenuously continues to disagree with that assertion. Holland clearly defines the terms to be different (as expounded in the James Kweeder (inventor) declaration filed on August 8, 2005). Shear-thinning fluids

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(sometimes referred to as *psedoplastic*) are those fluids whose viscosity decreases with increasing shear-rate. Thixotropic fluids, conversely, are those whose viscosity decreases with time under constant shear rate. Oldshue's classic text (attached herein for immediate reference) on fluid mixing supports this distinction.

Unfortunately, confusion has arisen on some of this terminology. The proper term for shear-thinning, *pseudoplastic*, has also come to mean a particular behavior of polymer melts (a Newtonian, non-Newtonian, and then again Newtonian viscosity). This ambiguity is nicely discussed in Holland. Consequently, the term shear-thinning has come to be used for the generic behavior of decreasing shear with increased shear rate. Examination of the Holland reference, particularly figures 1.20 and 1.21 (and the referencing text) illustrate the difference. *Shear-thinnable* refers to those liquids in which the viscosity decreases with increasing shear rate independent of time. *Thixotropic*, alternatively, describes a fluid in which the viscosity decreases with time while at constant shear-rate. While it is possible that a particular fluid be both thixotropic and shear-thinning (as illustrated in figure 1.21 of Holland), it is not required. A thixotropic viscosity can be independent of shear-rate (that is, Newtonian with respect to shear-rate but non-Newtonian with respect to time).

Further, it is certainly apparent that shear is required to bring-forth the thixotropic behavior of those fluids. Consequently, those inadequately skilled in the art have from time to time used the terms interchangably. *That usage – however - is grievously erroneous* and has been discussed by the Applicants in several documents submitted already in the case.

This lamentable ambiguity in terminology necessitates careful workers to define the meaning of their usage, as has been done in the current application. In this application, shear-thinning has been defined to mean decreasing viscosity with increasing shear-rate (see page 5, lines 33-35). Experimental evidence is also presented in the application to illustrate the meaning absolutely.

However, no such ambiguity exists with the term thixotropic. Thixotropic is defined strictly as time-dependent viscosity. While Hanke may chose to treat the terms "shear-thinnable"

and "thixotropic" interchangeably, the Applicant continues to point out that engineering literature (typified by Holland and Oldshue) most definitely does not support that usage.

Holland (and others) do point out that often thixotropic fluids are also shear-thinning (pseudoplastic) fluids. This common situation of time and shear-dependent viscosity gives rise to statements (like in Holland) of "in practice, thixotropic fluids are also shear-thinning". However, such statements do not guarantee that a thixotropic fluid is indeed shear-thinning. Further, such generalizations do not in any way change the definitions of the terms thusly erroneously rendering time-dependent and shear-dependent to mean the same thing.

A consequence of equating time and shear-dependence is the contention that shear is shear. Nothing can be further from the truth. As previously discussed in the James Kweeder declaration, shear-thinning requires device design with an eye toward generating the necessary shear-rate to achieve the desired reduction in viscosity. Thixotropy, conversely, requires attention to the necessary time to achieve viscosity reduction. Of course, a device designed for a fluid that is both shear-thinning and thixotropic will need to address both needs.

An excellent illustration of such sweeping over-extrapolation is the Examiner's analysis of Hoogendonk (US 3,083,406). In the examination, the assumption is made by the Examiner that since shear is being applied to the fluid at the roller-wall interface, consequently, the whole of the liquid volume is subjected to that shear and thusly capable of flowing under the centrifugal forces of the rotating prill cup. Implicit to that assumption is that the benefits of shear (argued as thixotropic by Hoogendonk) is realized throughout the system so long as it's available somewhere, anywhere in the system.

Further, despite the casual statement by Hoogendonk, it is by no means obvious that its inventive device functions because of thixotropy. Indeed, the '406 inventive device is an improvement on a previous device (US 3,055,049 on which Hoogendonk is also an inventor), which relies on a non-roller implementation to keep the inner walls of the prilling device clean. US 3,055,049 makes no claims nor arguments for the device advantageously modifying the fluid viscosity. Changing-out the fixed scraper for a roller-scraper does not modify the shear-mixing

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properties of the inventive device in any way. Consequently, the inventive device in US 3,083,406 is likely to be functional for the same reason that the inventive device was functional in US 3,055,049.

Further, as one skilled in the art and in light of definitively pressure-pumping devices disclosed in Frenken (US 3,988,398) and DE 2,355,660 (attached for immediate reference), one can argue that the inventive device in US 3,083,406 is functioning as a positive displacement, peristaltic pump that merely forces the reluctant fluid through the prilling orifice. Hoogendonk furthers enhances the positive displacement pump argument by then proposing pins on the rolling device to supplement peristaltic pumping action with a piston pumping action directly through the prilling orifice.

In US 3,083,406, Hoogendonk makes exactly one reference to thixotropy providing neither reference, experimental data, nor examples to support that argument. Given the thin zone of shear (the clearance between the roller and the wall) and other arguments for operability (US 3,055,049, US 3,988,398, DE 2,355,660), it is not obvious at all that thixotropy is responsible let alone that the inventive device is effective with shear-rate viscosity dependent fluids.

The difference between *shear-thinning* and *thixotropic* necessitates different approaches to device design. While exploiting either behavior requires shearing the material, it does not follow that one device suitable for one behavior will necessarily function as anticipated with the other behavior. In particular, *shear-thinning* usually requires that a particular shear-rate be achieved to accomplish the targeted viscosity. Consequently, it is necessary to specify that adequate shear-rate by the combination of shear device geometry and device velocity. *Thixotropy*, conversely, requires that shear be maintained for a specified time period. A device designed for shear-thinning may not sustain shear sufficiently long to process a thixotropic material and a device designed for thixotropy may not achieve adequate shear to process a shear-thinning material.

Hoogendonk Reference

Hoogendonk states in Column 1, lines 54-65 that: "In accordance with the present invention it has been found that prills of good quality can be obtained if the melt is sprayed from a reservoir which has a scraper arrangement including a rotary element rolling along the upright wall of the reservoir while the latter rotates. Owing to the rolling movement of the said element the inner wall of the reservoir remains clean and the spray openings do not become clogged. Due to the thixotropic properties of the melts to be sprayed, the shearing stresses produced by the rolling movement of the rotary element causes the melts to remain sufficiently fluid so that no solid material will deposit on the wall or in the spray openings."

Hoogendonk also states in Column 1, lines 35-42 that: "It is therefore an object of the present invention to provide an improved prilling device which prevents clogging of the spray openings and which has a relatively long life. A further object of the present invention is to provide an improved prilling device which utilizes a rolling member for preventing clogging of the spray openings." The assertion that the melts are fluid as based on thixotropic properties should be questioned. It has been asserted in the Kweeder Declaration filed on August 2, 2005 in this case that it may be the pressure created by the rollers, similar to the action of a positive peristaltic pump (see Exhibit B of this Declaration), causing the melts to prill easily over conventional methods used. In addition, if in fact the melts are fluid based on thixotropic properties, then the only part of the melt that is thixotropic is that part of the melt that is between the inner wall and the rollers. There is absolutely no teaching or suggestion in Hoogendonk that any portion of the remaining melt, such as that portion of the melt in the middle of the apparatus, is being swept by the rollers or thixotropic.

Hoogendonk mentions thixotropic fluids briefly without any discussion on these types of fluids or the shear-rate and/or time requirements when working with thixotropic fluids. Hoogendonk also does not suggest to one of ordinary skill in the art that there might be some benefit to sweeping and/or agitating the entire liquid volume in the prill head to shear-thin a

shear-thinnable mixture. If the agitator action of Hoogendonk does in fact work, it would be limited to the shear zone between the roller and the wall. The agitator action would not affect the entire liquid volume in the prill head.

Hoogendonk discloses a prilling device with an integral "agitator" that is specifically configured to roll along the inside wall of the prilling device for the specific purpose of keeping the prilling orifices "clear". One embodiment includes pins on the rolling device to mechanically clear the holes, the other is silent in whether a smooth roller or something textured is used to enhance clearing. (see Column 1, lines 59-66) But there is no teaching or suggestion that there is any benefit to using rollers to agitate or sweep the entire liquid volume of the reservoir, as mentioned in claim 1 of the above-referenced patent application.

Otsuka Reference

The Examiner should remove Otsuka et al. as a 35 USC §103(a) reference in this case. The Otsuka reference is filled with references to how difficult it is to take the molten materials described in Otsuka and process them by conventional prilling methods, including those that utilize agitators. (see Columns 5-6) Therefore, in order to combat the problems seen when incorporating the molten materials of Otsuka, Otsuka engineers using a series of mesh screens to break up and disperse the molten materials. After a fair reading of Otsuka, one of ordinary skill of the art would understand how to make molten materials containing nitrogen and potassium or phosphorus - but there is absolutely no suggestion or teaching that these molten compounds can be utilized by any conventional or modified prilling methods other than those described in the Otsuka reference, especially after reading Columns 5 and 6 of the reference. Therefore, combining this reference with another reference or references that describe prilling methods unlike those in Otsuka would be considered improper combination based on hindsight. The Applicant has respectfully requested that the Examiner review the Otsuka reference paying close attention to Columns 5 and 6 and withdraw this reference as a relevant reference in this matter.

Holland and Hanke References

The Holland and Hanke references merely disclose thixotropic and shear-thinning materials. There doesn't appear to be anything in either the Holland reference or the Hanke reference that cures the deficiencies of the Otsaka reference, the Hoogendonk reference or the Frenken reference, in combination with one or all of them, that would lead someone in the field of fertilizer chemistry and fertilizer production to prill a shear-thinnable mixture.

Bassetti Reference

The Bassetti reference discloses ammonium nitrate fertilizers and mixtures. There doesn't appear to be anything in the Bassetti reference that cures the deficiencies of the Otsaka reference, the Hoogendonk reference or the Frenken reference, in combination with one or all of them, that would lead someone in the field of fertilizer chemistry and fertilizer production to prill a shear-thinnable mixture.

ISSUE NO. 4 - §103 (A) REJECTION OF CLAIMS 16-17

Claim 1 recites:

"A method to prill a shear-thinnable mixture comprising the steps of:

a)   providing a molten first component;

b)   mixing at least a second component with said molten first component;

c)   reacting said components at a temperature and for a time sufficient to form a shear-thinnable mixture having a viscosity, whereby the viscosity decreases with increased shear rate;

d)   mechanically agitating said shear-thinnable mixture by an agitator in a prill head, wherein essentially the entire liquid volume in said prill head is swept by said agitator to shear thin said shear-thinnable mixture; and permitting said shear-thinned mixture to flow through holes in said prill head under the influence of a force selected from the group consisting of static pressure and centrifugal force.

THIXOTROPIC VERSUS SHEAR-THINNABLE

The Examiner has contended throughout the prosecution of this application that the terms "thixotropic" and "shear-thinnable" mean the same thing and are interchangeable. The terms *thixotropic* and *shear-thinnable*, while related, are not interchangeable and not understood in the field to mean the same thing.

Reference is made to Holland (1995) and Hanke (US 5,466,281) to support the assertion that thixotropic and shear-thinning are synonymous terms. The applicant strenuously continues to disagree with that assertion. Holland clearly defines the terms to be different (as expounded in the James Kweeder (inventor) declaration filed on August 8, 2005). Shear-thinning fluids

(sometimes referred to as *psedoplastic*) are those fluids whose viscosity decreases with increasing shear-rate. Thixotropic fluids, conversely, are those whose viscosity decreases with time under constant shear rate. Oldshue's classic text (attached herein for immediate reference) on fluid mixing supports this distinction.

Unfortunately, confusion has arisen on some of this terminology. The proper term for shear-thinning, *pseudoplastic*, has also come to mean a particular behavior of polymer melts (a Newtonian, non-Newtonian, and then again Newtonian viscosity). This ambiguity is nicely discussed in Holland. Consequently, the term shear-thinning has come to be used for the generic behavior of decreasing shear with increased shear rate. Examination of the Holland reference, particularly figures 1.20 and 1.21 (and the referencing text) illustrate the difference. *Shear-thinnable* refers to those liquids in which the viscosity decreases with increasing shear rate independent of time. *Thixotropic*, alternatively, describes a fluid in which the viscosity decreases with time while at constant shear-rate. While it is possible that a particular fluid be both thixotropic and shear-thinning (as illustrated in figure 1.21 of Holland), it is not required. A thixotropic viscosity can be independent of shear-rate (that is, Newtonian with respect to shear-rate but non-Newtonian with respect to time).

Further, it is certainly apparent that shear is required to bring-forth the thixotropic behavior of those fluids. Consequently, those inadequately skilled in the art have from time to time used the terms interchangably. *That usage – however - is grievously erroneous* and has been discussed by the Applicants in several documents submitted already in the case.

This lamentable ambiguity in terminology necessitates careful workers to define the meaning of their usage, as has been done in the current application. In this application, shear-thinning has been defined to mean decreasing viscosity with increasing shear-rate (see page 5, lines 33-35). Experimental evidence is also presented in the application to illustrate the meaning absolutely.

However, no such ambiguity exists with the term thixotropic. Thixotropic is defined strictly as time-dependent viscosity. While Hanke may chose to treat the terms "shear-thinnable"

27

and "thixotropic" interchangeably, the Applicant continues to point out that engineering literature (typified by Holland and Oldshue) most definitely does not support that usage.

Holland (and others) do point out that often thixotropic fluids are also shear-thinning (pseudoplastic) fluids. This common situation of time and shear-dependent viscosity gives rise to statements (like in Holland) of "in practice, thixotropic fluids are also shear-thinning". However, such statements do not guarantee that a thixotropic fluid is indeed shear-thinning. Further, such generalizations do not in any way change the definitions of the terms thusly erroneously rendering time-dependent and shear-dependent to mean the same thing.

A consequence of equating time and shear-dependence is the contention that shear is shear. Nothing can be further from the truth. As previously discussed in the James Kweeder declaration, shear-thinning requires device design with an eye toward generating the necessary shear-rate to achieve the desired reduction in viscosity. Thixotropy, conversely, requires attention to the necessary time to achieve viscosity reduction. Of course, a device designed for a fluid that is both shear-thinning and thixotropic will need to address both needs.

An excellent illustration of such sweeping over-extrapolation is the Examiner's analysis of Hoogendonk (US 3,083,406). In the examination, the assumption is made by the Examiner that since shear is being applied to the fluid at the roller-wall interface, consequently, the whole of the liquid volume is subjected to that shear and thusly capable of flowing under the centrifugal forces of the rotating prill cup. Implicit to that assumption is that the benefits of shear (argued as thixotropic by Hoogendonk) is realized throughout the system so long as it's available somewhere, anywhere in the system.

Further, despite the casual statement by Hoogendonk, it is by no means obvious that its inventive device functions because of thixotropy. Indeed, the '406 inventive device is an improvement on a previous device (US 3,055,049 on which Hoogendonk is also an inventor), which relies on a non-roller implementation to keep the inner walls of the prilling device clean. US 3,055,049 makes no claims nor arguments for the device advantageously modifying the fluid viscosity. Changing-out the fixed scraper for a roller-scraper does not modify the shear-mixing

properties of the inventive device in any way. Consequently, the inventive device in US 3,083,406 is likely to be functional for the same reason that the inventive device was functional in US 3,055,049.

Further, as one skilled in the art and in light of definitively pressure-pumping devices disclosed in Frenken (US 3,988,398) and DE 2,355,660 (attached for immediate reference), one can argue that the inventive device in US 3,083,406 is functioning as a positive displacement, peristaltic pump that merely forces the reluctant fluid through the prilling orifice. Hoogendonk furthers enhances the positive displacement pump argument by then proposing pins on the rolling device to supplement peristaltic pumping action with a piston pumping action directly through the prilling orifice.

In US 3,083,406, Hoogendonk makes exactly one reference to thixotropy providing neither reference, experimental data, nor examples to support that argument. Given the thin zone of shear (the clearance between the roller and the wall) and other arguments for operability (US 3,055,049, US 3,988,398, DE 2,355,660), it is not obvious at all that thixotropy is responsible let alone that the inventive device is effective with shear-rate viscosity dependent fluids.

The difference between *shear-thinning* and *thixotropic* necessitates different approaches to device design. While exploiting either behavior requires shearing the material, it does not follow that one device suitable for one behavior will necessarily function as anticipated with the other behavior. In particular, *shear-thinning* usually requires that a particular shear-rate be achieved to accomplish the targeted viscosity. Consequently, it is necessary to specify that adequate shear-rate by the combination of shear device geometry and device velocity. *Thixotropy*, conversely, requires that shear be maintained for a specified time period. A device designed for shear-thinning may not sustain shear sufficiently long to process a thixotropic material and a device designed for thixotropy may not achieve adequate shear to process a shear-thinning material.

Hoogendonk Reference

Hoogendonk states in Column 1, lines 54-65 that: "In accordance with the present invention it has been found that prills of good quality can be obtained if the melt is sprayed from a reservoir which has a scraper arrangement including a rotary element rolling along the upright wall of the reservoir while the latter rotates. Owing to the rolling movement of the said element the inner wall of the reservoir remains clean and the spray openings do not become clogged. Due to the thixotropic properties of the melts to be sprayed, the shearing stresses produced by the rolling movement of the rotary element causes the melts to remain sufficiently fluid so that no solid material will deposit on the wall or in the spray openings."

Hoogendonk also states in Column 1, lines 35-42 that: "It is therefore an object of the present invention to provide an improved prilling device which prevents clogging of the spray openings and which has a relatively long life. A further object of the present invention is to provide an improved prilling device which utilizes a rolling member for preventing clogging of the spray openings." The assertion that the melts are fluid as based on thixotropic properties should be questioned. It has been asserted in the Kweeder Declaration filed on August 2, 2005 in this case that it may be the pressure created by the rollers, similar to the action of a positive peristaltic pump (see Exhibit B of this Declaration), causing the melts to prill easily over conventional methods used. In addition, if in fact the melts are fluid based on thixotropic properties, then the only part of the melt that is thixotropic is that part of the melt that is between the inner wall and the rollers. There is absolutely no teaching or suggestion in Hoogendonk that any portion of the remaining melt, such as that portion of the melt in the middle of the apparatus, is being swept by the rollers or thixotropic.

Hoogendonk mentions thixotropic fluids briefly without any discussion on these types of fluids or the shear-rate and/or time requirements when working with thixotropic fluids. Hoogendonk also does not suggest to one of ordinary skill in the art that there might be some benefit to sweeping and/or agitating the entire liquid volume in the prill head to shear-thin a

shear-thinnable mixture. If the agitator action of Hoogendonk does in fact work, it would be limited to the shear zone between the roller and the wall. The agitator action would not affect the entire liquid volume in the prill head.

Hoogendonk discloses a prilling device with an integral "agitator" that is specifically configured to roll along the inside wall of the prilling device for the specific purpose of keeping the prilling orifices "clear". One embodiment includes pins on the rolling device to mechanically clear the holes, the other is silent in whether a smooth roller or something textured is used to enhance clearing. (see Column 1, lines 59-66) But there is no teaching or suggestion that there is any benefit to using rollers to agitate or sweep the entire liquid volume of the reservoir, as mentioned in claim 1 of the above-referenced patent application.

Otsuka Reference

The Examiner should remove Otsuka et al. as a 35 USC §103(a) reference in this case. The Otsuka reference is filled with references to how difficult it is to take the molten materials described in Otsuka and process them by conventional prilling methods, including those that utilize agitators. (see Columns 5-6) Therefore, in order to combat the problems seen when incorporating the molten materials of Otsuka, Otsuka engineers using a series of mesh screens to break up and disperse the molten materials. After a fair reading of Otsuka, one of ordinary skill of the art would understand how to make molten materials containing nitrogen and potassium or phosphorus - but there is absolutely no suggestion or teaching that these molten compounds can be utilized by any conventional or modified prilling methods other than those described in the Otsuka reference, especially after reading Columns 5 and 6 of the reference. Therefore, combining this reference with another reference or references that describe prilling methods unlike those in Otsuka would be considered improper combination based on hindsight. The Applicant has respectfully requested that the Examiner review the Otsuka reference paying close attention to Columns 5 and 6 and withdraw this reference as a relevant reference in this matter.

Holland and Hanke References

The Holland and Hanke references merely disclose thixotropic and shear-thinning materials. There doesn't appear to be anything in either the Holland reference or the Hanke reference that cures the deficiencies of the Otsaka reference, the Hoogendonk reference or the Frenken reference, in combination with one or all of them, that would lead someone in the field of fertilizer chemistry and fertilizer production to prill a shear-thinnable mixture.

Stengel Reference

The Stengel reference discloses ammonium nitrate fertilizers and mixtures. There doesn't appear to be anything in the Stengel reference that cures the deficiencies of the Otsaka reference, the Hoogendonk reference or the Frenken reference, in combination with one or all of them, that would lead someone in the field of fertilizer chemistry and fertilizer production to prill a shear-thinnable mixture.

Respectfully submitted,

Buchalter Nemer, A Professional Corp.

Dated: 5/30/2006   By:
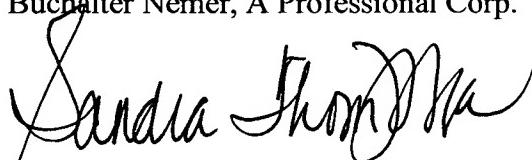
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APPENDIX OF PENDING CLAIMS

1.   (Previously Presented) A method to prill a shear-thinnable mixture comprising the steps of:

   a)   providing a molten first component;

   b)   mixing at least a second component with said molten first component;

   c)   reacting said components at a temperature and for a time sufficient to form a shear-thinnable mixture having a viscosity, whereby the viscosity decreases with increased shear rate;

   d)   mechanically agitating said shear-thinnable mixture by an agitator in a prill head, wherein essentially the entire liquid volume in said prill head is swept by said agitator to shear thin said shear-thinnable mixture; and permitting said shear-thinned mixture to flow through holes in said prill head under the influence of a force selected from the group consisting of static pressure and centrifugal force.

2.   (Original) The method according to claim 1 wherein said shear-thinnable mixture is a melt slurry.

3.   (Previously Presented) The method according to claim 1 wherein said first component is ammonium nitrate and said second component is ammonium sulfate.

4.   (Original) The method according to claim 1 wherein said shear-thinnable mixture comprises no more than about 2 weight percent water.

5.   (Original) The method according to claim 3 wherein said shear-thinnable mixture further comprises micronutrients.

6.   (Original) The method according to claim 1 wherein said prill head is one of a rotating bucket with a stationary blade, a stationary bucket with rotating scrappers and blades, and an agitated pressurized nozzle assembly.

7.   (Previously Presented) The method according to claim 1 wherein said prill head is wiped with surface-wiping blades.

8.   (Previously Presented) The method according to claim 7 wherein said first component is ammonium nitrate and said second component is ammonium sulfate.

9.   (Original) The method according to claim 7 wherein said shear-thinnable mixture comprises no more than about 2 weight percent water.

10.   (Original) The method according to claim 7 wherein said shear-thinnable mixture further comprises micronutrients.

Claims 11-14: Cancelled.

15.   (Previously Presented) The prilling method according to either claim 3 or claim 8, wherein the reaction time is about 10 minutes to about 15 minutes.

16.   (Previously Presented) The prilling method according to either claim 3 or claim 8, wherein the reaction temperature is at least about 180°C to about 200°C.

17.   (Previously Presented) The prilling method according to either claim 3 or claim 8, wherein the ammonium nitrate and the ammonium sulfate are present in equimolar amounts.

EVIDENCE APPENDIX

The attached Evidence was previously presented during prosecution of this case and is herein presented in this Appeal Brief:

Oldshue, James Y., Fluid Mixing Technology, Chemical Engineering (1983). Referred to first on page 6 of the Appeal Brief and admitted by the Examiner during prosecution.

DE 2355660, referred to on page 8 of the Appeal Brief and admitted by the Examiner during prosecution.

Declaration of James Kweeder, dated July 28, 2005 and admitted by the Examiner during prosecution (10/27/2005 Paper from Examiner).

FLUID
MIXING
TECHNOLOGY

James Y. Oldshue, Ph.D.

Vice President, Mixing Technology
Mixing Equipment Co., Inc.
Rochester, New York

CHEMICAL ENGINEERING

McGraw-Hill Publications Co., New York, N.Y.

Printed in the United States of America

FIFTEEN

VISCOUS MATERIALS AND NONNEWTONIAN BLENDING

INTRODUCTION

This chapter is concerned with high-viscosity blending of newtonian and nonnewtonian materials in the range of approximately 50,000 to 1 million cP (50 to 1000 Pa·sec) when measured at 5 sec^{-1} shear rate. It is also concerned with nonnewtonian materials in the intermediate range of 5000 to 50,000 cP (5 to 50 Pa·sec).

The definitions for *high-* or *intermediate*-viscosity ranges depend, to some extent, on scale. Since Reynolds number is a better measure of viscosity in the laminar range and part of the transitional range, the preceding definitions are appropriate for full-scale vessels 6 ft (1.8 m) or larger in diameter. For smaller vessels or pilot-plant equipment the definitions for both the high- and intermediate-viscosity ranges could easily be reduced by a factor of 10.

NONNEWTONIAN MATERIALS

Table 15-1 compares newtonian fluids with four types of nonnewtonian fluids. A fifth type, Bingham plastic, requires a minimum shear stress before any flow will occur in the material (Fig. 15-1).

Referring again to Table 15-1, there are two types of nonnewtonian behavior associated with shear rate. A *dilatant* material is one in which viscosity *increases* with shear rate (e.g., various concentrations of starch slurries exhibit this property). If viscosity *decreases* with shear rate, the material is *pseudoplastic*. This is the most common type of nonnewtonian fluid found in mixing applications.

Table 15-1 Definitions of nonnewtonian fluid behavior

Definition	Effect on viscosity μ of	
	Time increase	Shear rate increase
Newtonian	No effect	No effect
Nonnewtonian		
Pseudoplastic	No effect	Decrease
Dilatant	No effect	Increase
Thixotropic	Decrease	No effect
Rheopectic	Increase	No effect

Other kinds of nonnewtonian fluids are those which exhibit a change in viscosity with *time* at constant shear rate. If the viscosity *increases* with time, the material is *rheopectic*. If the viscosity *decreases* with time, the material is *thixotropic*. There are some popular terminologies that use the term *thixotropic* to refer to almost any nonnewtonian behavior. In this book, we use the separate terms *thixotropic* and *pseudoplastic* when referring to both time dependence and shear rate dependence.

Still another nonnewtonian behavior is called "viscoelastic." This is the property of developing flow normal to the major shear stress. Many materials that are
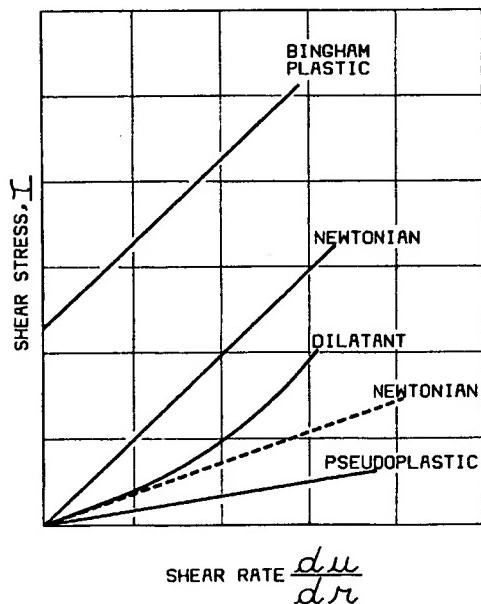
Figure 15-1 Shear stress vs shear rate for newtonian and nonnewtonian fluids.

FEDERAL REPUBLIC OF GERMANY
GERMAN PATENT OFFICE
PATENT NO. 2,355,660 A1
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C 05 C 13-00
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P 23 55 660.7-41

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METHOD AND APPARATUS FOR THE PRILLING OF FERTILIZER

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Examination request has been made according to Section 28b, Patent Law.

The invention concerns a method for the "prilling" of simple or mixed fertilizer, which mainly contains ammonium nitrate and its compounds, such as 20.5%, 26%, and 33% N and a

mixture of ammonium sulfate and nitrate. Furthermore, the invention concerns an apparatus for the carrying out of this method in plants with prilling towers.

The "prilling" of fertilizers, such as ammonium nitrate and urea in prilling towers, can be carried out in different ways—using mouthpieces (nozzles), rotary baskets, and prilling disks.

The first two possibilities, in which the product is thrown into the tower with a circular movement, require very large dimensions, so that the product does not strike the tower walls, under the effect of the centrifugal force. With the last-mentioned method, smaller towers can be used, because the product exits vertically. Herein lies one advantage of this method, since lower costs are generated. However, the mode of operation of these towers with the last-mentioned method involves a large number of problems--essentially in connection with those plants which produce mixed fertilizer with a low nitrogen content (20.5% to 27% N -ammonium nitrate limestone and a mixture of ammonium sulfate and nitrate), wherein additional solid, finely distributed nutrients are included in the suspension to be prilled.

For example, the scatter openings of the prilling disk on the tower head tend to become clogged, wherein the plant has to be shut down, in order to wash and clean the scatter openings. Since, on the other hand, the supply vessel of the prilling disk is so large, it must be insulated and heated, in order to maintain the suspension in the best operating conditions for the prilling disk. Furthermore, the fertilizer tends to adhere to the side walls of the supply vessel, since the suspension is hurled against the walls by the movements of the stirrer. In this way, solid sludge agglomerates are formed within the vessel, which fall down and clog the scatter openings. Also the great height of 1 - 1.15 m (40-45") requires the use of a stirrer with a very long, cantilever shaft, which causes considerable mechanical problems and leads to a very low work output.

As is known, many attempts have been undertaken to improve the operating conditions of such plants. It has not been possible thereby to prolong the operating times of the plants and to avoid their shutdowns. The reason for the failure of these attempts lies in the fact that two or three vessels are located on the prilling tower head, wherein one or two of these vessels serve as replacement vessels, which are cleaned and maintained, while another one is in operation. However, the short operating time of each vessel--sometimes only a few hours--does not permit a continuous mode of operation.

Accordingly, the goal of the invention is to create an actually effective possibility for the prilling of simple or mixed fertilizer, wherein the work is done vertically. A continuous operation is to be made possible, in which, using a single supply vessel, a cleaning of the prilling disk or of the supply vessel is not required.

To attain this goal, the invention creates a method for the prilling of simple or mixed fertilizer in plants with prilling towers, which is characterized in that pure ammonium nitrate or a mixture of ammonium nitrate with other nutrients, such as ammonium sulfate or pulverized

limestone, in various combinations, with a water concentration, which can attain values below 0.5%, is prilled vertically.

In accordance with the invention, it is possible to use towers of smaller dimensions. Thus, a yield of approximately 400 tons/day (17 tons/day [sic; tons/h] in 20.5% N) is attained with a 5 x 5-m tower, which would be impossible with the same dimensions using rotary baskets and mouthpieces. A plant working in accordance with the invention, with a capacity of 300 tons/day, has already been in operation for a year and has not had to be shutdown a single time up to now because of the failure of the prilling apparatus--without it being necessary to exert any external effects, such as, perhaps, hammer blows or vibrating systems. Various fertilizers were thereby produced--from ammonium nitrate to ammonium nitrate limestone and a mixture of ammonium sulfate and nitrate.

Other advantages and features of the invention can be deduced from the following description of a preferred exemplified embodiment, in connection with the appended design. The figures of the design show the following:

Figure 1, a scheme of a plant, which delivers an increased output with operation in accordance with the invention;

Figure 2, a side view of the supply vessel located on the tower head;

Figure 3, a sketch of the supply vessel according to Figure 2;

Figure 4, a vertical section through the supply vessel according to Figure 2.

In accordance with Figure 1, a mixing vessel 4 is supplied, via a conduit 1, with an aqueous solution with 83% ammonium nitrate or another concentration. Through a conduit 2, finely pulverized limestone reaches the mixing vessel 4. Furthermore, a conduit 3 is provided, through which additional components, such as iron sulfate (discharged from the screen of the finished product) or ammonia, are added. The supply of the mixed vessel 4 takes place continuously and in adapted quantities, to obtain various nitrate types present on the market--from pure ammonium nitrate to the weakest fertilizers (20.5%). In order to attain a uniform and homogeneous and thorough mixture of the various components, the mixing vessel 4 is equipped with a stirrer. Furthermore, it is heated, so as to attain the correct operating temperature. The temperature of the mixture must be kept at values above the melting point of the ammonium nitrate, wherein one remains as close as possible to this temperature, so as to avoid reactions of the mixture.

It was found that the temperature is preferably between approximately 80° and approximately 150°C, wherein unburned limestone of such a particle size is used that 100% passes through a screen with 70 Tyler mesh.

The suspension coming from the mixing vessel 4 arrives at a pump 6 via a conduit 5 and from there to a filter 8 via a conduit 7. The filter 8 is used to remove nonuniform parts of the

suspension. From the filter 8, the suspension arrives at a film evaporator head 10 via a conduit 9. This works with a descending or sloping film, as is described, for example, in US-PS 2,089,945. A solution which contains a compound and a volatile liquid flows in counterflow to a heated inert gas as a continuous film over a heated surface. The latter is maintained at a temperature which lies above the melting point of the compound in an anhydrous state. The flow rate of the liquid is such that the film comes into contact with the surface only briefly, so as to avoid a decomposition of the compound. The contact time, however, is sufficient to dehydrate the suspension.

Since the suspension flows downward as a thin film, its water content is reduced, in stages, from the entry point into the evaporator head to the evaporator bottom, where an essentially anhydrous product is obtained. From the evaporator bottom, the suspension arrives at a supply vessel 12 of the prilling tower 13 via a conduit 11. The residence time of the suspension in the supply vessel 12 should be as short as possible, because the product leaves the bottom of the evaporator 10 with little water and has a temperature which is close to that of sludge solidification. The supply vessel 12 is equipped with a stirrer, so that the suspension goes through a prilling disk located on the bottom of the supply vessel 12. The supply vessel is concentric to the prilling tower 13, which is supplied with air, which enters in the vicinity of its bottom. The air migrates, in counterflow to the prill-shaped product, through the head of the prilling tower.

The prilling tower 13 has the shape of a quadrangular prism with a triangular, prism-shaped bottom. One of the edges between the base surfaces is open and supplies a conduit 14, which leads to a cooler 15. Here, the product is cooled, before it arrives at a screen 16 and subsequently at a coating drum 17. The product is conveyed to the storage building via the conduit 18. The geometric shape of the prilling tower is not a critical feature. Thus, the prilling tower can have a quadrangular, rectangular, or round cross-section.

In connection with Figures 2, 3, and 4, a few experiments should be explained, which were made so as to improve the mode of operation of the disks and the prilling. The first supply vessels were round, had a stirrer, and measured 800-1200 mm in height. Their diameters were between 650 and 750 nm. The prilling disk was fixed on the vessel bottom by a flange. In order to eliminate the cause for the defective mode of operation of these arrangements, different modification and improvement experiments were undertaken. Thus, for example, a hammer striking the prilling disk from below was used, which was changed later with a hammer striking from above, in order not to allow the sludge in the boreholes to solidify and to clog the prilling disk. Furthermore, the number (4, 6, and 8) of stirring blades and their angle of inclination was changed, so as to give the suspension at the entry of the prilling disk an appropriate and rapid movement. Also, a chamber was provided in the area of the stirring blades, which was bounded

below by the prilling disk and above by a perforated disk, so as to prevent sludge from being hurled against the vessel walls and in this way, from forming solid agglomerates which clogged the prilling disk. Another experiment consisted in providing a Teflon sleeve connected with the inner vessel walls by a crosspiece, so as to eliminate the mechanical deficiencies of the stirrer shaft, which were caused by its length. The prilling disk had between 3000 and 7000 boreholes with various diameters. If, on the other hand, no clogging occurs, and the product remains in the vessel only a short time, then this shaft can be designed much smaller and lower, wherein the mode of operation of the stirrer is improved and its mechanical deficiencies are eliminated.

The mode of operation of the stirring blades was similar to a sieving machine (passe-vite). The stirring blades forced the sludge to flow through the boreholes, wherein drops were formed on the outside of the boreholes, which covered the entire surface of the disk. Under the effect of the force of gravity, the drops fell, in counterflow to the air rising through the prilling tower, downward. The product obtained in this manner was very nonuniform and contained a large fraction of reject coarse grains and sometimes, such large agglomerates that they did not solidify over the entire height of the prilling tower. On the other hand, a few drops solidified before becoming detached from the prilling disk, so that the latter became clogged.

In accordance with the invention, on the other hand, the stirrer works as an impeller, similar to that of a centrifugal pump and throws the product through boreholes provided only on the circumference of the prilling disk--at a prespecified initial speed, which depends on the blade speed and on the air flow rising in the prilling tower. The adjustment of these variables--namely, blade speed, borehole diameter, and supply flow--thus makes possible a better quality control of the finished product with respect to the size and shape of the grains, because, namely, the dropping of the product is independent of its weight, its viscosity, and the higher or lower efficiency of the vibration system.

Since the stirrer-impeller exerts a pressure on the sludge on the entry into the boreholes, one can a) enlarge the borehole diameter; b) avoid clogging; c) produce smaller fine grains; d) increase the production capacity with the same output.

With the stirrers working in a manner similar to a sieving machine, the sludge which does not pass through the boreholes has a tendency to rise upwards on the vessel walls under the pressure effect of the blades. On the other hand, if one uses a stirrer-impeller, in accordance with the invention, which works in a low cylinder chamber supplied in the middle part of the blades with the sludge to be prilled, the sludge does not rise on the walls, since all the sludge is driven by the blades through the boreholes.

The apparatus in accordance with the invention, as it is represented in Figures 2, 3, and 4, therefore offers the possibility of attaining good results, without the disadvantages explained above appearing.

In accordance with the drawing, a truncated cone-shaped supply vessel 19 is provided, whose upper larger base 20 has a diameter of approximately 350 mm. The lower, smaller base 21 is open and connected with a chamber 22 for the stirring blades. This chamber has the shape of a cylinder with a height of approximately 50 mm and a diameter of approximately 600 mm. Its underside consists of a prilling disk 23, which is affixed to a flange. The entire arrangement is affixed, using flaps 24 on beams of the tower head and, as a whole, has a height of approximately 300 mm and a weight of approximately 110 kg. It is made of stainless steel and has metal supports for 25 reinforcement.

The suspension coming from the evaporator 10 enters laterally through an inlet 26 into the supply vessel. A stirrer shaft 27, screwed on the blade system, is located in the axis of the truncated cone. A stopper 28 is used to introduce ammonium to control the pH value of the suspension--in view of ammonium losses which occur in the evaporator 10. The prilling disk has around 300 boreholes, which have a diameter of approximately 2 mm and are located on the circumference.

The described arrangement has proven good in the production of any product, without having to undertake possible modifications. The quantity of the product produced is dependent on the speed of the impeller (impeller shaft coupled with an adjustable speed-range electric motor), on the level of product in the supply vessel, and on the diameter of the boreholes in the prilling disk. The changing of the prilling disk can be carried out extraordinarily quickly (about half an hour), since the entire arrangement can be handled very easily.

In order to clarify the efficiency of the method in accordance with the invention, the following table shows some examples from the production area of an industrial plant--determined in various seasons:

2355660

① PRODUKT	② GEMISCH			⑥ Stündlich-Herstellung	⑦ Gesamtes Harnstoff	⑧ KORNGRÖSSEANALYSE				
	③ Ammon Nitrat als 100%	④ Gebrochener Kalkstein	⑤ Ammon Sulfat			4 mm %	2,8-4 mm %	2-2,8 mm %	1-2 mm %	1 mm %
Ammonsulfat-salpeter 26% N ⑨	38%	-	62%	7 t	26,2%	0,5	30,1	35,0	29,2	5,2
Ammonsulfat-salpeter 30% N ⑨	65%	-	35%	4 t	30,2%	-	14,5	33,3	49,5	2,7
Nitrokalkstein 20,5% N ⑩	60%	40%	-	12 t	20,5%	0,2	10,8	30,0	56,4	2,6
Nitrokalkstein 26% N ⑩	76%	24%	-	10 t	26%	0	6,0	29,9	58,9	5,2
Ammonsalpeter 33,5% N ⑪	98%	2%	-	10 t	33,8%	0,2	10,9	34,5	49,1	5,3

⑫ Die Korngrösseanalyse betriff das zum Lagerhaus gesandten Produkt nach dieses zwischen 8-10% von verworfenen Grobkörnern und 10-12% von verworfenen Feinkörnern im Siebe gelassen hat welche in das Mischgefäss rückgeführt sind.

509820/0915

[Key to previous page:]

1 Product
2 Mixture
3 Ammonium nitrate as 100%
4 Broken limestone
5 Ammonium sulfate
6 Hourly production
7 Total urea
8 Particle size analysis
9 Mixture of ammonium nitrate and sulfate
10 Nitro-limestone
11 Ammonium nitrate
12 The particle size analysis concerns the product sent to the storage building after it has left between 8-10% rejected coarse grains and 10-12% rejected fine grains in screens, which are returned to the mixing vessel.

Claims

1. Method for the prilling of simple or mixed fertilizer in plants with prilling towers, characterized in that pure ammonium nitrate or a mixture of ammonium nitrate with other nutrients, such as ammonium sulfate or pulverized limestone, in various combinations, with a water concentration which can reach values below 0.5%, is prilled vertically.

2. Method according to Claim 1, characterized in that a truncated cone-shaped supply vessel is connected to a cylindrical chamber with its smaller base.

3. Method according to Claims 1 or 2, characterized in that the suspension to be prilled enters the cylindrical chamber via the supply vessel.

4. Method according to one of Claims 1 - 3, characterized in that the suspension to be prilled is thoroughly stirred by a stirrer, whose blades rotate within the cylindrical chamber.

5. Method according to one of Claims 1 - 4, characterized in that the bottom of the cylindrical chamber consists of a disk, whose boreholes are located in two concentric circles on the circumference.

6. Method according to one of Claims 1 - 5, characterized in that the stirrer works as an impeller.

7. Method according to one of Claims 1 - 6, characterized in that the suspension is kept at a prespecified pressure by speed regulation of the blades of the stirrer-impeller.

8. Method according to one of Claims 1 - 7, characterized in that the blade plane is inclined at an angle to the horizontal, in order to maintain a prespecified suspension pressure at the entry into the prilling disk, which is adapted to the production conditions resulting from the system.

9. Method according to one of Claims 1 - 8, characterized in that the suspension to be prilled contains ammonium nitrate.

10. Method according to one of Claims 1 - 9, characterized in that the suspension consists of ammonium nitrate with one or more solid and finely distributed nutrients.

andere

zermahltes
Kalkstein

83% iges
Ammonsalpeter

10 Verdampfer

12 Priller

13

Prill-
turm

9

2

3

1

5

4'

Pumpe 7

Filter

8

6

Kühler   Sieb   Lageru

15   16   17

14   18

Zumischungs
gefäss

Überzug
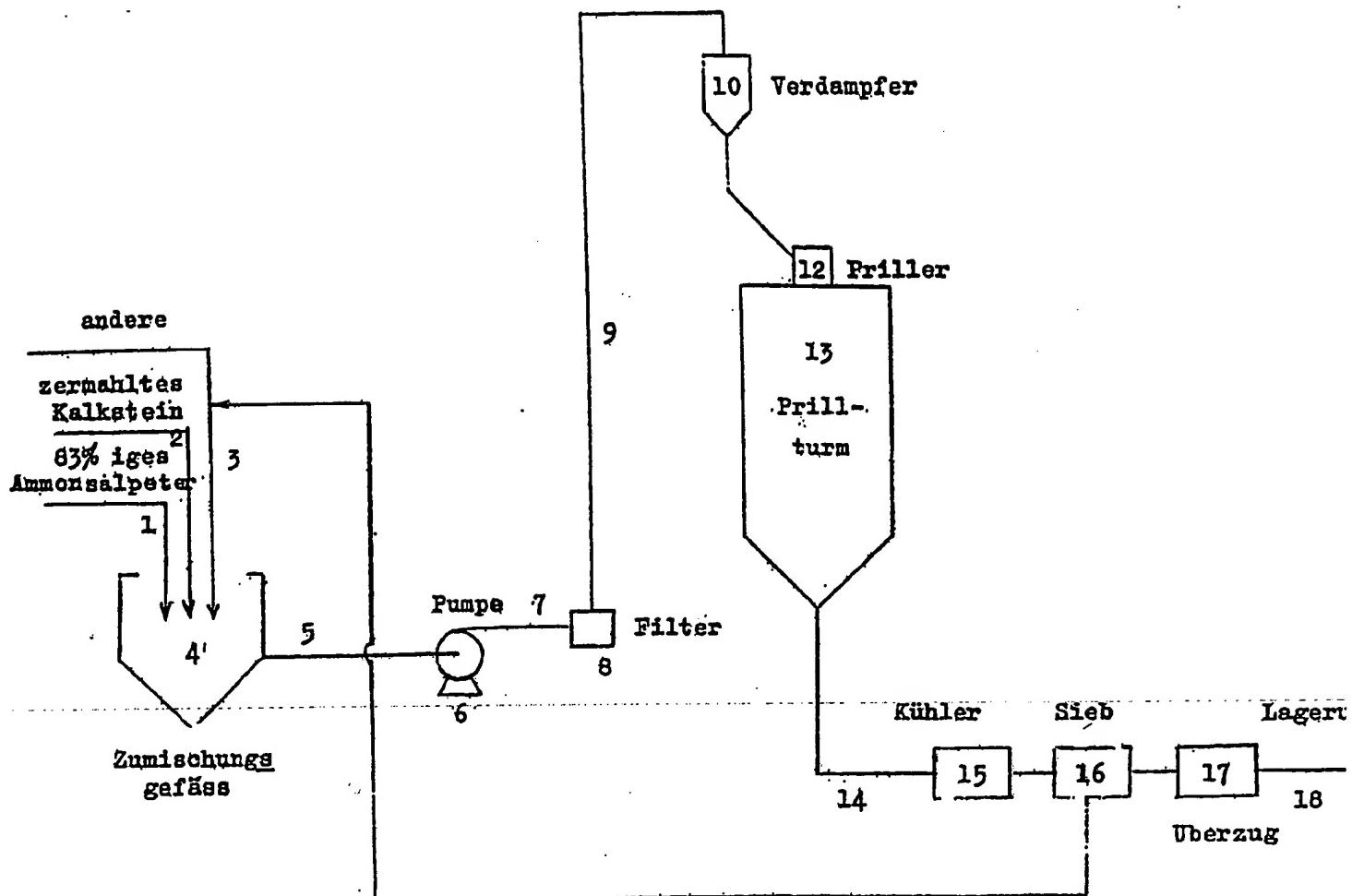
Figure 1

Key: 1   83% Ammonium nitrate
     2   Pulverized limestone
     3   Other
     4   Admixture vessel
     6   Pump
    10   Evaporator
    12   Priller
    13   Prilling tower
    15   Cooler
    16   Screen
    17   Coating
    18   Storage
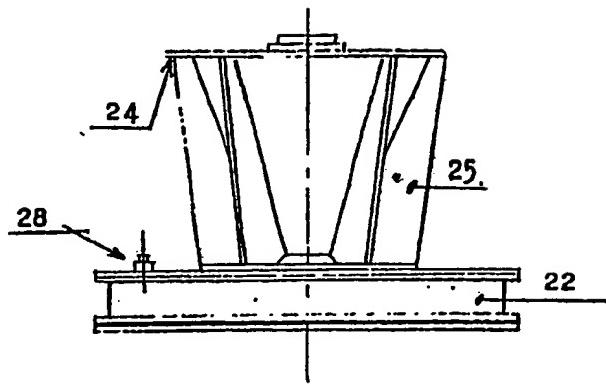
Figure 2
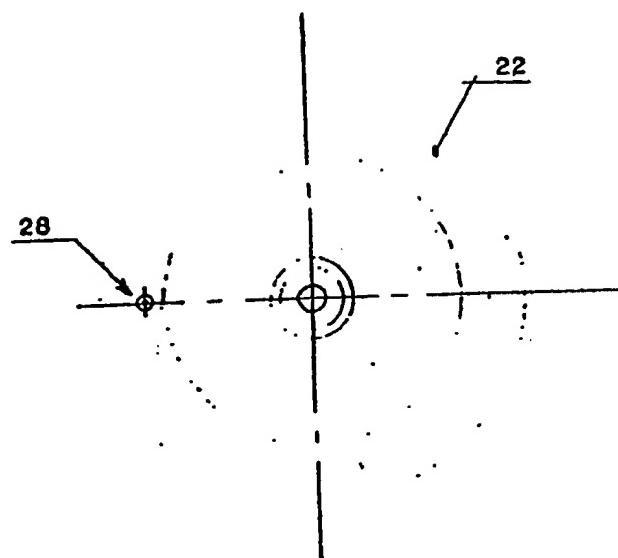
Figure 3
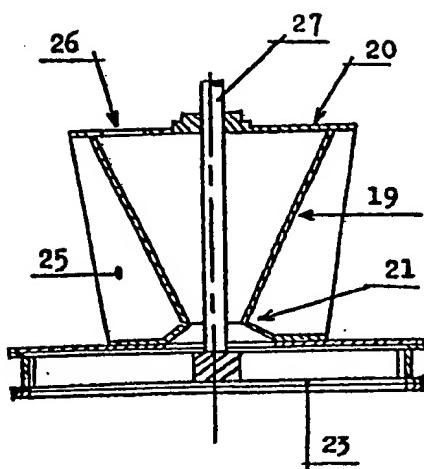
Figure 4

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor:	**Kweeder et al.**	Examiner:	**Robert A. Madsen**
Serial No:	**09/468,668**	Art Unit:	**1761**
Filed:	**December 21, 1999**		
For:	**PRILLING METHOD**		

DECLARATION OF JAMES KWEEDER
UNDER 35 USC §1.132

I, the undersigned, James Kweeder, hereby declare as follows:

1. My educational and professional qualifications are shown in the attached resume (see Exhibit A).

2. Further to my work in connection with the prosecution of the above-referenced patent application, I have read and I understand the text of the following documents and issued patents (hereafter referred to as "cited art"):

    a. Hoogendonk (US 3083406)

    b. Frenken et al (US 3988398)

    c. Otsuka et al (US 3529326)

    d. Holland et al., Fluid Flow for Chemical Engineers, pp. 52, 53 and 55, (1995)

    e. Hanke et al (US 5466281)

    f. Bassetti et al (US 5378259)

    g. Stengel (US 3021207)

3. It is my understanding from reading and reviewing the cited art that none of these references separately teach:

> A method to prill a shear-thinnable mixture comprising the steps of:
>
> providing a molten first component;
>
> mixing at least a second component with said molten first component;
>
> reacting said components at a temperature and for a time sufficient to form a shear-thinnable mixture having a viscosity, whereby the viscosity decreases with increased shear rate;
>
> mechanically agitating said shear-thinnable mixture by an agitator in a prill head, wherein essentially the entire liquid volume in said prill head is swept by said agitator to shear thin said shear-thinnable mixture; and permitting said shear-thinned mixture to flow through holes in said prill head under the influence of a force selected from the group consisting of static pressure and centrifugal force.

4. Respectfully submits that the terms *thixotropic* and *shear-thinnable*, while related, are not interchangeable and not understood in the field to mean the same thing. Examination of the Holland reference, particularly figures 1.20 and 1.21 (and the referencing text) illustrate the difference. *Shear-thinnable* refers to those liquids in which the viscosity decreases with increasing shear rate independent of time. *Thixotropic*, alternatively, describes a fluid in which the viscosity decreases with time while at constant shear-rate. While it is possible that a particular fluid be both thixotropic and shear-thinning (as illustrated in figure 1.21 of Holland), it is not required. A thixotropic viscosity can be independent of shear-rate (that is, Newtonian with respect to shear-rate but non-Newtonian with respect to time).

5. The difference between *shear-thinning* and *thixotropic* necessitates different approaches to device design. While exploiting either behavior requires shearing the material, it does not follow that one device suitable for one behavior will necessarily function as anticipated with the other behavior. In particular, *shear-thinning* usually requires that a particular shear-rate be achieved to accomplish the targeted viscosity. Consequently, it is necessary to specify that adequate shear-rate by the combination of shear device geometry and device velocity. *Thixotropy*, conversely, requires that shear be maintained for a specified time period. A device designed for shear-thinning may not sustain shear sufficiently long to process a thixotropic material and a device designed for thixotropy may not achieve adequate shear to process a shear-thinning material.

6. As one who is skilled in the art of fertilizer chemistry and fertilizer production, it would not be obvious to me, after a fair reading of each member of the cited art and after reviewing the combined cited art, to prill a shear-thinnable mixture, as listed in paragraph 3, because of the following:

Hoogendonk Reference

a. Hoogendonk states in Column 1, lines 54-65 that:

"In accordance with the present invention it has been found that prills of good quality can be obtained if the melt is sprayed from a reservoir which has a scraper arrangement including a rotary element rolling along the upright wall of the reservoir while the latter rotates. Owing to the rolling movement of the said element the inner wall of the reservoir remains clean and the spray openings do not become clogged. Due to the thixotropic properties of the melts to be sprayed, the shearing stresses produced by the rolling movement of the rotary element causes the melts to remain sufficiently fluid so that no solid material will deposit on the wall or in the spray openings."

b. As I read the above paragraph in the Hoogendonk reference, I first question the assertion that the melts are fluid as based on thixotropic properties. I assert that it may be the pressure created by the rollers, similar to the action of a positive peristaltic pump (see Exhibit B), causing the melts to prill easily over conventional methods used.

c. In addition, as I read the above paragraph, if in fact the melts are fluid based on thixotropic properties, then as I - one of ordinary skill in the art - understand it, the only part of the melt that is thixotropic is that part of the melt that is between the inner wall and the rollers. There is absolutely no teaching or suggestion in Hoogendonk that any portion of the remaining melt, such as that portion of the melt in the middle of the apparatus, is being swept by the rollers or thixotropic.

d. Hoogendonk states in Column 1, lines 35-42 that: "It is therefore an object of the present invention to provide an improved prilling device which prevents clogging of the spray openings and which has a relatively long life. A further object of the present invention is to provide an improved prilling device which utilizes a rolling member for preventing clogging of the spray openings."

e. Hoogendonk mentions thixotropic fluids briefly without any discussion on these types of fluids or the shear-rate and/or time requirements when working with thixotropic fluids.

f. Hoogendonk also does not suggest to one of ordinary skill in the art that there might be some benefit to sweeping and/or agitating the entire liquid volume in the prill head to shear thin a shear-thinnable mixture.

g. If the agitator action of Hoogendonk does in fact work, it would be limited to the shear zone between the roller and the wall. The agitator action would not affect the entire liquid volume in the prill head.

h.   Hoogendonk discloses a prilling device with an integral "agitator" that is specifically configured to roll along the inside wall of the prilling device for the specific purpose of keeping the prilling orifices "clear". One embodiment includes pins on the rolling device to mechanically clear the holes, the other is silent in whether a smooth roller or something textured is used to enhance clearing. (see Column 1, lines 59-66) But there is no teaching or suggestion that there is any benefit to using rollers to agitate or sweep the entire liquid volume of the reservoir, as mentioned in claim 1 of the above-referenced patent application.

Frenken Reference

a.   The Frenken device includes a pump impeller with the specific purpose of raising the interior fluid pressure and forcing the melt to flow through the holes. The above-referenced patent application very specifically states that we prill through a combination of static and/or centrifugal pressure and not via pressurization.

b.   The Frenken reference, like the Hoogendonk reference, is not placing any importance or critical embodiment on sweeping and/or agitating essentially the entire liquid volume in the reservoir to shear-thin a shear-thinnable liquid. In Column 2 of the reference, lines 14-23, it is explicitly stated that the distance from the inner wall of the reservoir to the ends of the blades **is not critical**.

c.   As a comparison, DE 2355660 teaches a cylindrical chamber with stirring blades, similar to that described in Frenken. However, the DE 2355660 points out that modifying the configuration of the chamber to be similar to the one described in the above-mentioned patent application would result in thickening, clogging of the prill holes, nonuniform product, large fraction of reject course grains and occasional large agglomerates that did not solidify in the prill tower.

Otsuka Reference

a.   The Otsuka reference is filled with references to how difficult it is to take the molten materials described in Otsuka and process them by conventional prilling methods, including those that utilize agitators. (see Columns 5-6)

b.   In order to combat the problems seen when incorporating the molten materials of Otsuka, Otsuka engineers using a series of mesh screens to break up and disperse the molten materials.

c.   After a fair reading of Otsuka, I would understand how to produce molten materials containing nitrogen and potassium or phosphorus - but I would not understand how these molten compounds can be utilized by any conventional or modified prilling methods other than those described in the Otsuka reference, especially after reading Columns 5 and 6 of the reference.

Holland and Hanke References

The Holland and Hanke references merely disclose thixotropic and shear-thinning materials. There doesn't appear to be anything in either the Holland reference or the Hanke reference that cures the deficiencies of the Otsaka reference, the Hoogendonk reference or the Frenkern reference, in combination with one or all of them, that would lead someone in the field of fertilizer chemistry and fertilizer production to prill a shear-thinnable mixture.

Bassetti and Stengel References

The Bassetti and Stengel references disclose ammonium nitrate fertilizers and mixtures. There doesn't appear to be anything in either the Bassetti reference or the Stengel reference that cures the deficiencies of the Otsaka reference, the Hoogendonk reference or the Frenken reference, in combination with one or all of them, that would lead someone in the field of fertilizer chemistry and fertilizer production to prill a shear-thinnable mixture.

I hereby declare that all statements made herein of my own knowledge are true and that statements made on information or belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Title 18, United States Code, Section 1001, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Executed at Chester, Virginia, this 28 day of July, 2005.

By: _____

James Kweeder, Ph.D.

Exhibit A

Curriculum Vitae

James A. Kweeder, Ph.D.
Principal Research Engineer
Honeywell Nylon LLC

Education:

Rose-Hulman Institute of Technology, Terre Haute, IN: Bachelor of Science, Chemical Engineering

Clarkson University, Potsdam, NY: Master of Science, Chemical Engineering. Thesis: *Evaporation Control in Float-Zone Refining of Cadmium Telluride.*

Clarkson University, Potsdam, NY: Doctor of Philosophy, Chemical Engineering. Dissertation: *Nucleation Mechanisms in Microcellular Polymer Foams.*

Publications:

US Patent 6,689,181: *Non-explosive ammonium sulfate nitrate composite materials as fertilizers,* (2004).

A hypothesis for nucleation in conventional and microcellular foams. Kweeder, J. A.; Ramesh, N. S.; Rasmussen, D.; Campbell, G. A., Foams '99, International Conference on Thermoplastic Foam, 1st, Parsippany, NJ, United States, Oct. 19-20, 1999 (1999)

Nucleation mechanisms in microcellular polymer foams. Dissertation, Kweeder, James A.. Clarkson Univ., Potsdam, NY, USA. (1997),

US Patent 5,414,154: *Reduction of methylbenzofuran impurity in phenol,* (1995),

An experimental study on the nucleation of microcellular foams in high-impact polystyrene. Ramesh, N. S.; Kweeder, J. A.; Rasmussen, D. H.; Campbell, G. A., Annual Technical Conference - Society of Plastics Engineers (1992).

The nucleation of microcellular polystyrene foam. Kweeder, J. A.; Ramesh, N. S.; Campbell, G. A.; Rasmussen, D. H., Annual Technical Conference - Society of Plastics Engineers (1991),

Related Experience:

Honeywell (formerly AlliedSignal)
Research Engineer, Monomer Technology (1991 to 1997)
Principal Research Engineer, Monomer Technology (1997 to present)

Principal investigator for product and process development for the production of caprolactam (the monomer for Nylon-6) and related co-products and by-products. Chemicals include phenol, acetone, alpha-methyl styrene, cyclohexanone, cyclohexanol, cyclohexanone oxime, ammonium carbonate, ammonium nitrite, hydroxylamine, ammonium sulfate, adipic acid.

Exhibit B

Positive Displacement Pumps for Agricultural Applications[1]

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The primary function of a pump is to transfer energy from a power source to a fluid, and as a result, to create lift, flow or greater pressure on the fluid. A pump can impart three types of hydraulic energy to any fluid: lift, pressure, and velocity.

The classification of pumps used in this publication first defines the principle by which energy is added to the fluid, then identifies the means by which this principle is implemented, and finally, distinguishes among specific pump geometries commonly used. Under this classification system, all pumps may be divided into two major categories:

1. **dynamic pumps**, where continuously added energy increases the velocity of the fluid which is later converted to lift or pressure, and

2. **positive displacement pumps**, where periodically added energy directly increases pressure or lift.

This publication will only discuss positive displacement pumps. These pumps are normally used to produce high fluid pressures which are necessary for numerous agricultural applications; among them, the injection of chemicals into a pressurized irrigation pipe system. Other applications include fluid transfers, sprayers, and chemical metering. Positive displacement pumps used for injection of chemicals in agricultural irrigation systems are discussed in more detail than other pumps. Dynamic pumps used for pumping water in

agricultural applications are discussed in another publication.

Positive displacement pumps are self-priming, which is especially advantageous when handling hazardous chemicals. It is important to note that self-priming does not imply that air can be pumped against pressure or into a pressurized system to prime a pump. Rather, the discharge line should be vented to the atmosphere until all air is removed from the system during priming. This is important if the suction line is long, contains large quantities of air, or if the pump is discharging into a pressurized system upon starting. The pump is primed when all air is displaced by liquid in the suction line and there is a continuous liquid column in the discharge line.

Positive displacement pumps described in this publication can be subdivided into reciprocating and rotary pumps (Figure 1). This classification refers to whether a reciprocating or rotating mechanism is used to transfer energy to the fluid.

RECIPROCATING PUMPS

A reciprocating pump is one in which a piston or a diaphragm displaces a given volume of liquid with each stroke. The change in internal volume of the pump creates the high pressure which forces liquid into the discharge pipe. Check valves on both the suction and the discharge sides of the pump allow the pumped liquid to flow in one direction only.

Figure 1. Types of positive displacement pumps.

Figure 2b. Piston pump - discharge stroke.
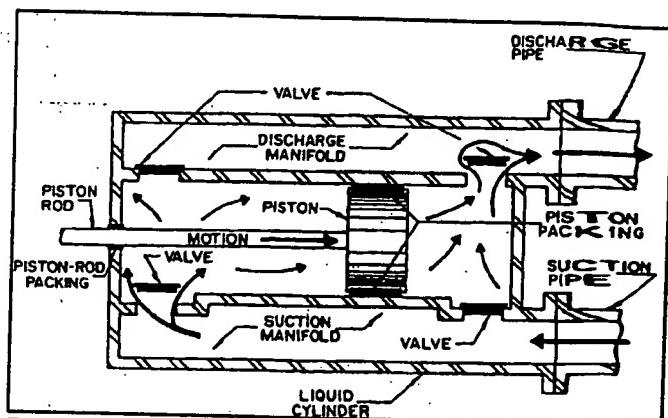

Figure 2c. Double acting piston pump.

Figure 2a. Piston pump - suction stroke.

Reciprocating pumps are classified as piston or diaphragm pumps. This classification is based on the type of reciprocating element used to transfer energy to the fluid.

Piston Pumps

In piston pumps, a piston, which is attached to a mechanical linkage, transforms the rotary motion of a drive wheel into the reciprocating motion of the piston. On an intake stroke (Figure 2a) the liquid enters the cylinder through the suction check valve. On a compression stroke (Figure 2b) it is forced into the discharge line through the discharge check valve. This action is similar to the action of a piston in the cylinder of an automobile engine.

The flow rate of a simple piston pump is not constant since there is no flow on the intake stroke and the flow varies from zero to a maximum and back to zero on each compression stroke.

Pulsation of flow can be reduced by using a double action piston pump (Figure 2c) where the volume on both sides of the piston is used for pumping liquid. In this case, each suction stroke is a companion compression stroke for the opposite side
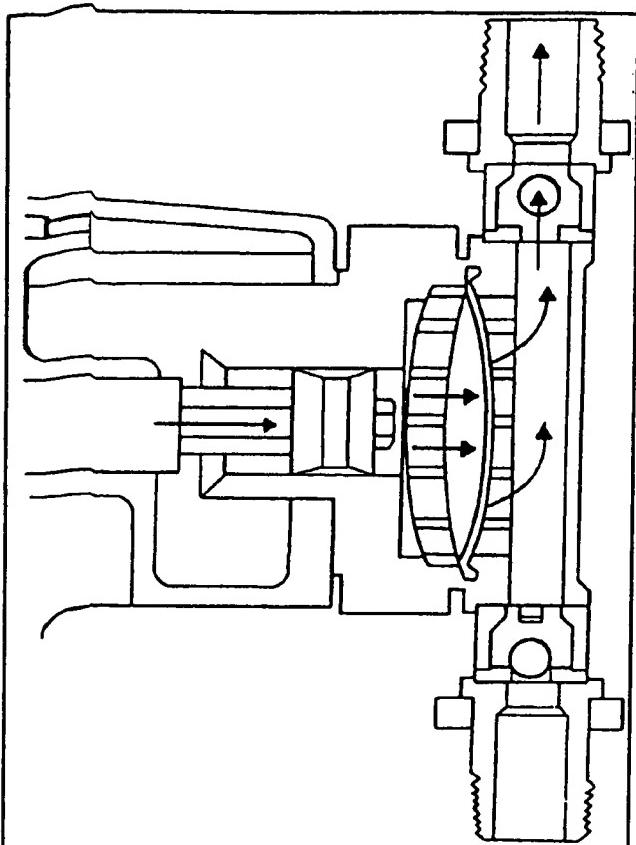
Figure 3a. Diaphragm pump - suction stroke.

Figure 3b. Diaphragm pump - discharge stroke.

of the pump and the liquid is pumped on both stroke.

To reduce the fluctuations even further, more than one cylinder can be employed in the same pump. In addition, each cylinder can be single or double acting. However, it should be noted that the pulsation of the flow is usually not a problem when chemicals are injected into an irrigation system, and pumps of the type shown in Figure 2a and Figure 2b are commonly used for this purpose.

The flow rate of a piston pump can be varied by changing the reciprocating speed of the piston or the length of the piston stroke. Variable speed drive motors are also used sometimes to alter pumping rates, but this is a more expensive option. The upper limit for metering pumps of this kind is about 350 strokes per minute. Capacities of piston pumps vary from a few cubic inches per hour to 20 gpm. For chemical injection into irrigation systems, capacities in the range of 0.01 to 0.5 gpm are commonly used.

Piston pumps can create high pressures (up to 50,000 psi) and they deliver a constant flow rate independent of the discharge pressure. They can run dry without damage and do not require downstream check valves. Piston pumps are mechanically simple and can have an exceptionally long life if properly maintained. Maintenance is critical, however, because internal parts of the pump are in direct contact with the pumped liquid. This may create problems when corrosive chemicals are being pumped. In addition, piston pumps cannot be used for pumping abrasive liquids or chemicals which crystalize.

The cost of piston pumps ranges from several hundred to 2 or 3 thousand dollars. The more expensive models can be adjusted for calibration while operating, while the simpler models must be stopped for any adjustment. Piston pumps are bulky, heavy, and can have a significantly pulsating discharge.

Diaphragm Pumps

Diaphragm pumps are the most common positive displacement pumps used in agricultural applications. The operation of a diaphragm pump is similar to that of a piston pump. The pulsating motion is transmitted to the diaphragm through a fluid or a mechanical drive, and then through the diaphragm to the pumped liquid. A schematic of this type of pump is presented in Figure 3a and Figure 3b.

A major advantage of diaphragm pumps is that the pumped liquid does not come into contact with most of the working parts of the pump. The only moving parts which are in contact with the pumped liquid are the diaphragm and the suction and discharge check valves. Hence, diaphragm pumps are suitable for pumping corrosive liquids which is often the case with chemical injection into an irrigation system.

For diaphragm pumps, the pumping characteristics depend on the method by which the driving force is transmitted to the diaphragm. Less expensive ($200 - $500) diaphragm pumps are mechanically driven. They use an unsupported diaphragm which is moved in the discharge direction by a cam or by a piston. These mechanically driven diaphragm pumps have pressure limitations of 125 to 150 psi and capacity limits of 12 to 15 gpm.

The pumping rate for mechanically driven diaphragm pumps vary with the pressure that the pumps are operating against. Thus, field calibration while pumping against a pressurized system is required for accurate calibration. However, diaphragm pumps are made to be adjustable while operating, which makes the process of calibration easier.

Since most irrigation systems operate at a constant, predetermined pressure, mechanical diaphragm pumps are often used for chemical injection into an irrigation system. Once calibrated for the operating pressure, the pump does not have to be frequently adjusted.

Liquid-driven diaphragm pumps provide all of the advantages of both piston pumps and mechanically driven diaphragm pumps. They can create the same high pressures as piston pumps. For liquid-driven diaphragm pumps, the pumping rate is also independent of the discharge pressure (as in piston pumps) since for all practical purposes liquid is incompressible. The discharge capacity of these pumps is up to 20 gpm, but rates of 0.01 to 0.5 gpm are most common for chemical injection applications.

Liquid-driven diaphragm pumps are usually significantly more expensive ($1,500 - $3,000) than mechanically driven diaphragm pumps and, for some applications, this additional expense cannot be justified. Liquid-driven diaphragm pumps are often used when high precision is required such as for injection of pesticides into irrigation systems.

Diaphragm pumps can be run dry for extended periods of time without damage. However, operation against a closed discharge must be avoided since high pressure may build up on a discharge side. Shutoff valves, responding to high pressure on the discharge side, should be installed on the suction side of the pump.

Most diaphragm pumps can be adjusted for calibration when running. If the length of stroke can be adjusted using a mechanical linkage, they do not require variable speed drives to alter pump discharge.

ROTARY PUMPS

Rotary pumps transfer liquid from suction to discharge through the action of rotating gears, lobes, vanes, screws, or similar mechanisms. These rotating elements operate inside a rigid casing. Rotary pumps do not require check valves for proper operation. However, these pumps are often equipped with check valves to assist with priming and to avoid backward flow when the pump is stopped.

Rotary pumps can be classified into the following groups (Figure 1): gear, lobe, vane, screw, flexible impeller. This classification is based on the type of rotating element used to transfer energy to the fluid.

Gear Pumps

A gear pump can be classified as internal (Figure 4) or external (Figure 5) depending on the position of the gears. In the internal gear pump (Figure 4) the inside gear is attached to the pump drive shaft and the outside gear, which is a part of the pump casing, is an idler gear. In the external gear pump there are two meshing gears of equal size (Figure 5) located in the pump case. One of the gears is attached to the pump shaft and the other is an idler gear.
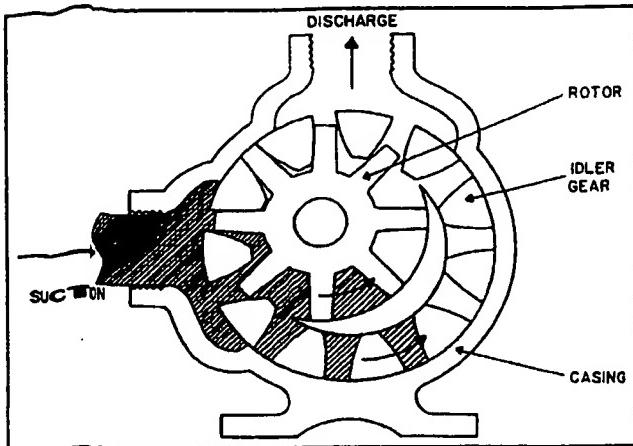
Figure 4. Internal gear pump.
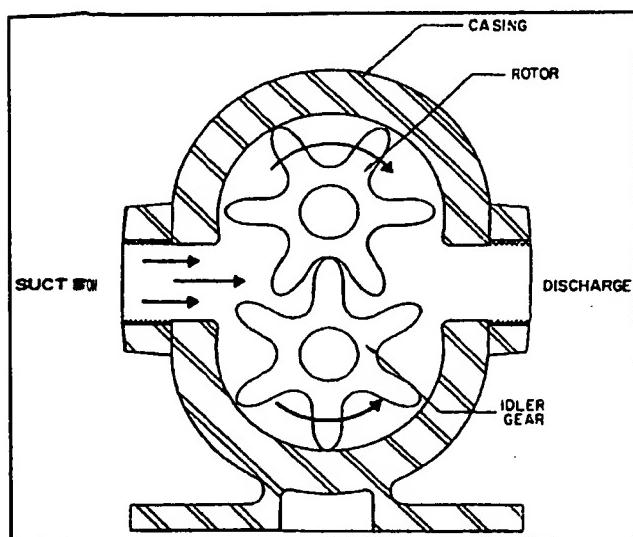
Figure 5. External gear pump.

The operation of a gear pump is based on the partial vacuum which is created by the unmeshing of the rotating gears. This vacuum causes liquid to flow into the pump. Then, the liquid is carried between the gears and the casing to the discharge side of the pump. The meshing of the rotating gears on the discharge side prohibits backward flow and generates an increase in pressure which forces liquid into the outlet line.

Both internal and external gear pumps can theoretically be run in either direction, but in most cases they are equipped with pressure relief valves which prevent buildup of pressure above safe levels. The recommended direction of flow is clearly marked on the outside of the pumps. Check valves are
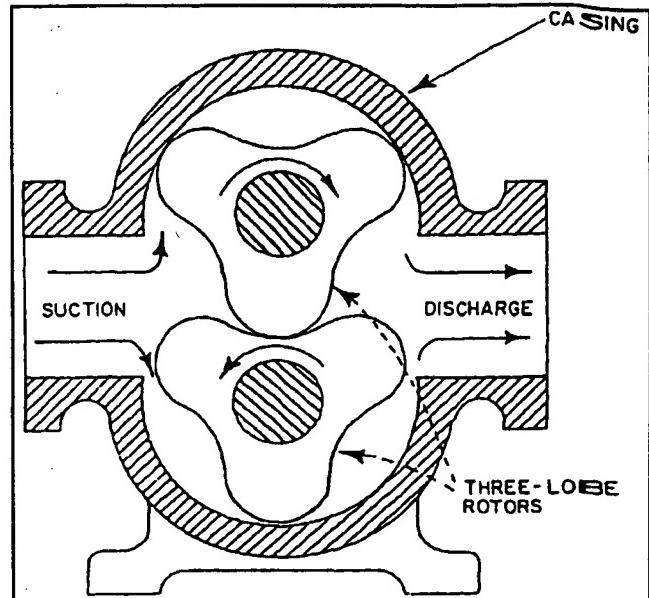
Figure 6. Lobe pump with two impellers.

sometimes also used to insure proper direction of flow.

Gear pumps produce a constant discharge for a set speed of rotation, thus the pulsation of flow is negligible. They depend on the pumped liquid for lubrication and they can be damaged when they run dry. Gear pumps can easily be damaged when operating against a closed discharge and, therefore, a pressure relief valve is a necessary part of the pump installation. A bypass valve, which returns part of the liquid from the discharge to the suction side of the pump when the pressure is too high, is used by some manufacturers to prevent pump damage.

The alignment of internal parts in a gear pump is very critical because close clearances between moving parts are essential. As a result, abrasive fluids quickly damage these pumps. Gear pumps should be used for chemical injection of nonabrasive liquids only. They are also used in some hydraulic servo systems, sprayers, and as pumps for machine-tool services.

Lobe Pumps

Lobe pumps operate like external gear pumps, but the gears are replaced by impellers which have two or three lobes. Figure 6 illustrates a three-lobe pump with two impellers. The number of lobes will determine the amount of pulsation from the pump output. The greater the number of lobes, the more constant is the discharge from the pump.

Figure 7. Vane pump.
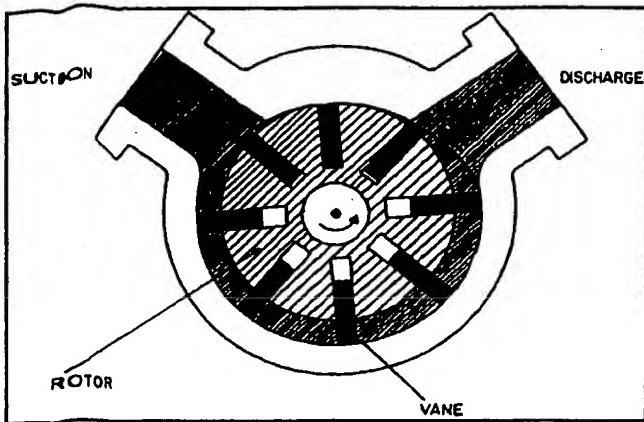
Figure 8. Single screw pump.

The lobed impellers are easier to replace and tend to wear less with abrasives than the gears in the gear pump. The alignment is also less critical in the lobe pump than in the gear pump.

Lobe pumps are self-priming and can pump liquid which contains vapor or air. For these reasons lobe pumps are frequently used in vacuum pumps and compressors.

Vane Pumps

In vane pumps, fluid is pumped using a rotor mounted off-center in the pump casing. Rectangular vanes are placed at regular intervals around the rotor, and the vanes are free to move in a slot (Figure 7). As the rotor spins, the vanes are moved toward the casing by centrifugal force, and they form chambers in which the fluid is moved along the casing. Liquid enters the pump due to the vacuum which is created by the eccentricity (off-center location) of the rotor when in operation. The same eccentricity creates the pressure at the outlet.

Vane pumps produce a constant flow rate for a given rotor speed. The pulsation is negligible and the original capacity is not affected until the vanes are significantly worn. These pumps can be operated in either direction. Like most of the positive displacement pumps, vane pumps cannot operate against a closed discharge without damage to the pump. Because of this, pressure relief valves are often installed on the discharge side of the pump and check valves are used to establish direction of flow.

Vane pumps are frequently used for lubrication of machine-tools, and in electro-hydraulic control systems. They are also used in some spraying equipment.

Screw Pumps

Screw pumps consist of helical screws which revolve in a fixed casing (Figure 8). As the screw rotates in the casing, a cavity created between the screw and the casing progresses towards the discharge side of the pump. This movement creates a partial vacuum which draws liquid into the pump. The shape of the casing at the discharge end is such that the cavity becomes closed. This generates pressure, pushing the liquid into the discharge line. Some screw pumps use double screws which guide the liquid to the same discharge point.

Screw pumps produce a constant discharge with negligible pulsation. They have an exceptionally long life expectancy. They are built in a very wide range of sizes and capacities with pressure ranges from 50 to 5000 psi. A pressure relief valve is required at the installation since screw pumps cannot be operated against a closed discharge. Screw pumps can operate in one direction only.

Significant disadvantages of screw pumps are that they are bulky and heavy. Application of screw pumps in agriculture is limited to food processing and hydraulic systems for machine tools.
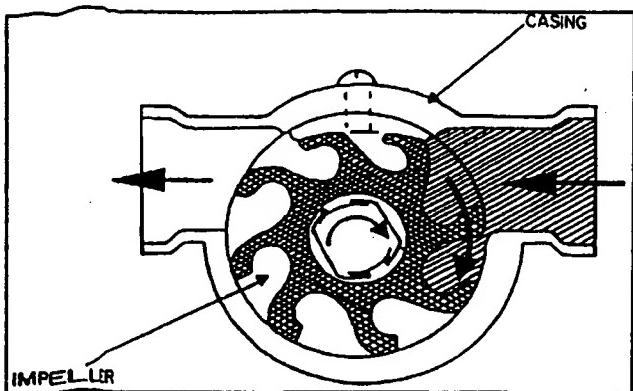
Figure 9. Flexible impeller pump.

Flexible Impeller Pumps

Flexible impeller pumps consist of flexible-bladed impellers which are placed eccentrically in casings (Figure 9). The impeller blades unfold as they pass the suction port, creating a partial vacuum which causes liquid to flow into the pump. As the rotor moves, the blades bend due to the eccentric placement of the rotor, resulting in a squeezing action on the liquid and increased pressure. The discharge of a flexible impeller pump is thus uniform with negligible pulsation.

Flexible impeller pumps are typically not equipped with pressure relief or check valves, because they can operate against a closed discharge for a short time without damage and the contact between the impeller and casing prevents backwards flow. These pumps cannot pump against high pressure and are usually used in applications where the pressure does not exceed 30 psi. Because of pressure limitations, the flexible impeller pumps are not usually selected for the injection of chemicals into irrigation systems. They are commonly used as fluid transfer and low pressure metering pumps.

MISCELLANEOUS PUMPS

Eccentric cam pumps and peristaltic pumps are discussed here. They differ from the previously discussed rotary pumps by the fact that the liquid being pumped is not in direct contact with a rotating element.
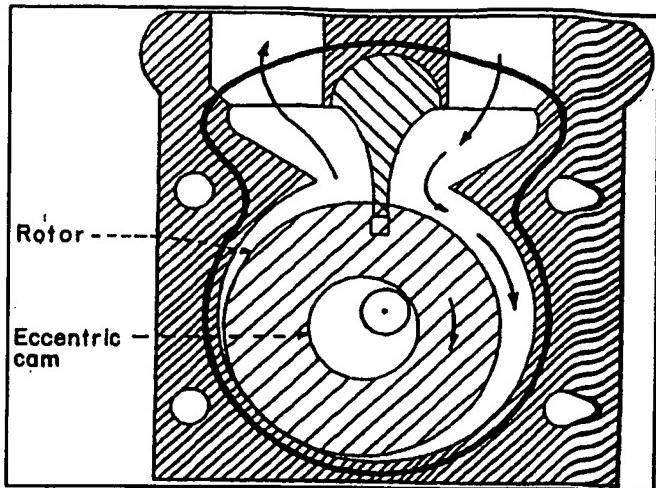

Figure 10. Eccentric cam pump.

Eccentric Cam Pumps

Eccentric cam pumps are also called rotating piston or plunger pumps (Figure 10). These pumps have some of the characteristics of both rotary and reciprocating pumps. The primary component of these pumps is the eccentric cam which rotates within a circular housing inside a cylindrical plunger which is in direct contact with the pumped liquid. Cam rotation causes the cylindrical plunger to change position relative to the fixed casing. The cavity created between the plunger and the housing transmits the fluid towards the pump discharge. During the cam cycle the volume available for liquid inside the pump progressively decreases, resulting in increased pressure at the discharge side of the pump.

Eccentric cam pumps are self-priming, do not require check valves and can pump in either direction. These pumps have their primary moving parts separated from the pumped liquid, and therefore can be used for pumping corrosive substances.

There are other variations of cam pumps, such as: diaphragm cam pumps, flexible liner cam pumps, and variable volume eccentric cam pumps (also called sliding shoe pumps) which are sometimes used for agricultural applications. More information on these pumps can be found in Holland and Chapman (1966).
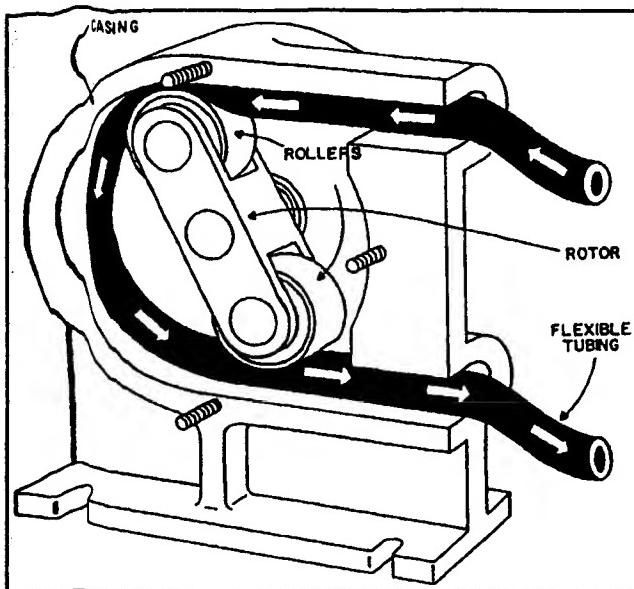
Figure 11. Peristaltic pump.

Peristaltic Pumps

Figure 11 shows a typical peristaltic pump. A flexible tube passes through the fixed casing of the pump. A rotor with rollers attached to it moves and presses against the flexible tube. This squeezing action produces an even flow of liquid. For proper action it is important that the tubing is flexible enough to allow the rollers to squeeze it until it is completely closed. Special tubing, often tygon tubing, is used with peristaltic pumps. Because rollers continuously pass over and compress it, the tubing life expectancy is limited. This life varies with the type of tubing, but averages about 200 hours.

Peristaltic pumps are self-priming. They do not require check valves. The pumped fluid is completely isolated from the moving parts, which permits pumping of corrosive substances. These pumps can be run dry for extended periods of time without damage to the pump.

Peristaltic pumps are used mostly in chemical laboratories, but they can be used for injection of chemicals into small irrigation systems. Their capacity is limited and most of them produce relatively low pressure (30-40 psi). However, special models are manufactured which can produce up to 100 psi.

OPERATING PROBLEMS

Common problems that occur during operation of positive displacement pumps are presented in Table 1. Six common problems and possible reasons for each one are addressed in this table:

1. the pump does not deliver any liquid,
2. the pump delivers less liquid than its rated capacity,
3. the prime is lost during operation of the pump,
4. the pump is noisy,
5. the pump wears more rapidly than should be expected, and
6. the pump takes too much power.

SUMMARY

Different types of positive displacement pumps are discussed in this publication. Positive displacement pumps are classified as reciprocating and rotary types depending on the mechanism used to transfer energy to the fluid. Reciprocating pumps, including both diaphragm and piston types, are commonly used for chemical injection into agricultural irrigation systems. Rotary pumps include gear, lobe, vane, screw, flexible impeller, eccentric cam and peristaltic pumps. Basic principles of operation, typical applications, advantages and disadvantages of each type are presented.

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Hydraulic Handbook. 1979. Colt Industries, Fairanks Morse Pump Division, Kansas City, Kansas.

Karassik I. J., W. C. Krutzsch, W. H. Fraser, J. P. Messina. 1976. *Pump Handbook*. McGraw-Hill Book Company, New York.

Positive Displacement Pumps for Agricultural Applications

Table 1. Problems with operation of positive displacement pumps.

No liquid delivered	Pump delivers less than rated capacity	Loss of prime while pump is operating	Pump is noisy	Rapid pump wear	Pump takes too much power
2. Insufficient NPSHa	2. Insufficient NPSHa	2. Air leak develops in pump or seal	2. Misalignment	2. Grit or abrasive material in liquid	2. Shaft packing too tight
4. End of suction line in water	4. Wear on pump leads to increased clearances and slip	4. Liquid vaporizes in suction line	4. Bent rotor shaft (for rotary pumps)	4. Corrosion	4. Misalignment
6. Relief valve jammed open	6. Relief valve jammed open				6. Discharge line too small
8. Suction or discharge valves closed	8. Suction of discharge valves partially closed				
	10. Liquid viscosity differs from that specified in pump selection				

RELATED APPEALS AND INTERFERENCES APPENDIX

There are no other appeals or interferences in this matter known to appellant, and therefore, there are no additional attachments under this heading.